National Educators' Workshop: Update 88

Standard Experiments in Engineering Materials Science and Technology

NASA

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Standard Experiments in Engineering Materials Science and Technology

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PREFACE

Many new and modified materials continue to emerge on the engineering and technology scene. Those responsible for educating the technical personnel who will be charged with keeping the United States world competitive must ensure that materials science, engineering, and technology courses develop competencies in the emerging materials without forsaking traditional engineering materials.

NEW Update 86 brought together leaders from industry and education to learn new materials and to develop strategies for integrating them into engineering materials technology instruction. These leaders concluded that educators need new laboratory experiments and class demonstrations to expose students to the nature and properties of the new engineering materials.

NEW Updates 86, 87, and 88 made progress in improving teaching in engineering materials, science and technology by presenting information to educators on

> Materials in Manufacturing
> Recent Developments in Metallurgy
> Metallic Coatings and Surface Modification
> Advances in Polymeric Materials
> Adhesives Bonding and Interface Technology
> Service Life Prediction of Organic Materials
> Developments in Optical Fibers
> Electronic Materials Development
> Advances in Composite Materials
> Ceramic Materials
> Long-Range Macro-Materials Cost-Estimating
> Microcomputers for Design and Analysis
> Modular Units for Teaching

The goal of *NEW Update 88* was to obtain and to share with the educational community a large number of experiments relating to the nature and properties of engineering materials that cover the field. Further, we sought single experiments that encompassed a variety of

concepts while not requiring elaborate or expensive setups as they focus on newer materials.

Hopefully, the experiments and ideas presented in this publication provide useful information to assist in teaching about materials in a manner that will excite students and motivate them to probe for the facts and wonders related to all materials.

NEW Update 88 and this printing resulted from considerable cooperative efforts by individuals in government, private industry, and education. While the goal of putting together a comprehensive manual of experiments for the ever emerging field of materials science has yet to be fulfilled, this collection represents a good effort in that direction. The workshop organizers express their appreciation to all those who assisted and hope this material is useful to you.

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CONTENTS

HIGH T_C SUPERCONDUCTORS: ARE THEY MAGNETIC?

Dr. Robert D. Shull
Institute for Materials Science and Engineering
National Institute of Standards and Technology

OBJECTIVE: To demonstrate the magnetic characteristics of the new superconducting oxides with transition temperatures above liquid nitrogen temperature (77 K).

EQUIPMENT: Liquid nitrogen, a disc (plus a partial disc) and a rod of the superconducting oxide $YBa_2Cu_3O_{7-x}$ ($0<x<0.5$), a strong cylindrical tube magnet, a small (0.1g) samarium cobalt magnet, a Styrofoam cup, a ring stand, and plastic tweezers. (Another good project to precede this one would be to make your own $YBa_2Cu_3O_{7-x}$ superconductors from powders of barium carbonate, yttrium oxide, and copper oxide.)

PROCEDURE: (1) First find out whether $YBa_2Cu_3O_{7-x}$ is magnetic at liquid nitrogen temperature and if so, what the sign of its magnetic susceptibility is. Tie a string around the middle of the rod of $YBa_2Cu_3O_{7-x}$ and suspend it from a ring stand; partially fill a Styrofoam cup with liquid nitrogen and immerse the rod in the liquid by slipping the cup around it from underneath.

****

SAFETY WARNING: LIQUID NITROGEN IS EXTREMELY COLD AND CAN EASILY FREEZE THE WATER IN YOUR BODY. THEREFORE, WEAR GLOVES WHEN HANDLING

****

Lower the cup and quickly bring the tubular magnet (because its field lines are convex with respect to the center) close to the cold rod (the magnet's axis pointing toward the rod) and observe the rotation of the sample. If the long axis of the rod is parallel to the axis of the magnet, the rod has a negative susceptibility (i.e., diamagnetic); and if it lines up perpendicular to the magnet's axis, the rod has a positive magnetic susceptibility (i.e., it is paramagnetic or ferromagnetic). You may need to perform this test several times so that you can be sure that your observation is being made while the $YB_2Cu_3O_{7-x}$ is still colder than 90 K (this material's superconducting transition temperature, T_c, above which the material is NOT superconducting).

(2) When a material becomes a superconductor it will exclude all magnetic flux that exists in it before the transition. This is called the Meissner effect and is shown schematically in Figure 1. In this figure the magnetic field (H) lines are shown by arrows. Note that no lines penetrate the superconducting sphere at temperatures below T_c when the field intensity is less than the critical field value, H_c. (Actually, H_c, the field value above which the material also loses its superconductivity, is temperature dependent: equal to zero at T_c and increasing in value very quickly with decreasing temperature.) Such a phenomenon results from the creation of circulating electrical currents inside the superconductor to completely oppose the external field, thereby also creating a repulsive force. If the external field is created by a permanent magnet, the repulsive force may be strong enough

to actually move the magnet. Figure 2A shows just such an experiment to test this possibility for $YBa_2Cu_3O_{7-x}$. Turn the Styrofoam cup upside down and on top of it place the superconductor disc. On top of the superconductor place the small samarium cobalt magnet and then pour liquid nitrogen around the superconductor (the small rim on the cup's bottom will contain the liquid) until it reaches 77 K (you can tell when this occurs by the large decrease in bubbling of the liquid). What does the magnet do? Can you push it around with the tweezers?


Normal;
$T > T_c$
or $H > H_c$

Superconducting;
$T < T_c$
or $H < H_c$
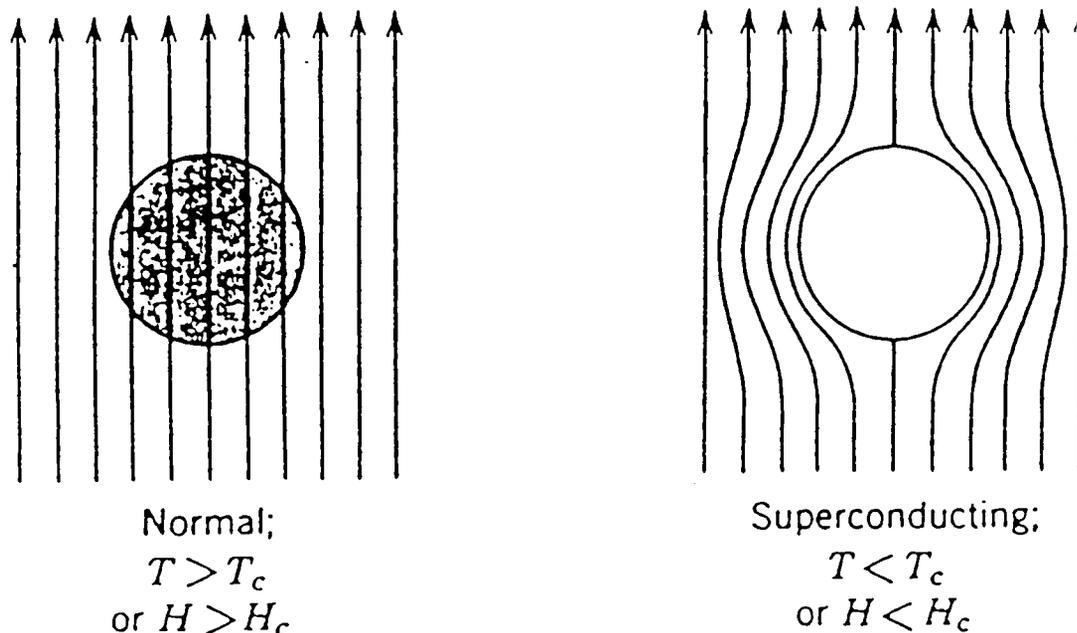
FIGURE 1

(3) Actually, the high T_c superconducting oxides, like $YBa_2Cu_3O_{7-x}$, are Type II superconductors: that is, they allow some magnetic flux lines into the material when the field exceeds a critical field value slightly lower than H_c, and the material remains partially superconducting to field values much larger than H_c. If these flux lines get trapped, i.e., pinned in the material, the magnitude of the supercurrents will decrease as less field is being opposed. Consequently, for materials like this, it might be expected that the levitation experiment just performed would be dependent upon whether or not there was a magnetic field present when the material was cooled to below T_c. The experiment depicted in Figure 2B tests this possibility; perform it by cooling the $YBa_2Cu_3O_{7-x}$ disc to 77 K and then place the magnet on top. This is a test of the "magnetic shielding" of the superconductor. Does the magnet levitate now? Is there any difference between the two experiments shown in Figure 2? Look at the magnetic hysteresis loops for this material measured at two different temperatures shown in Figure 3 to explain these differences (or lack of differences) observed. From these hysteresis loops, note that they enclose more area the lower the temperature of measurement; recall that H_c increases from zero with decreasing temperature below T_c. (An interesting question arises from Figure 3B: after applying a field of 3000 Oe and reducing it to zero, there is a net positive magnetization from the trapped flux remaining. If the small magnet were now brought close to this superconductor, would there actually be an attraction rather than a repulsion?)

(4) During either experiment depicted in Figure 2, could the magnet be pushed to the side of the disc and still remain in the air? Repeat the experiments shown in Figure 2, but this time use the incomplete disc of $YBa_2Cu_3O_{7-x}$. Does anything different occur? Why? What is the difference between pushing the magnet near the edge of the complete disc and putting the magnet in the center of the partial disc?

"HIGH" TEMPERATURE
SUPERCONDUCTING OXIDES

(A)

Superconducting Disc
Magnet

300 K

77 K

(B)

300 K

77 K
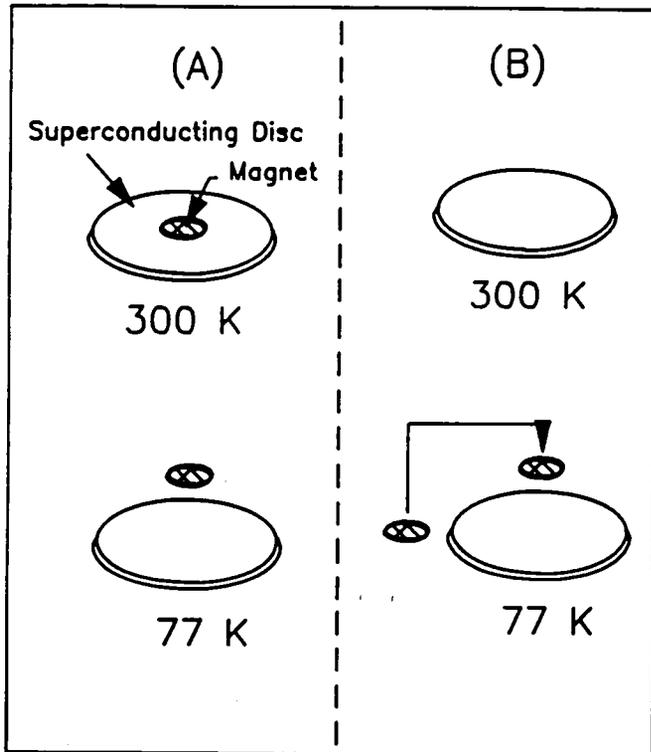
MAGNETIC LEVITATION EXPERIMENTS

Figure 2

Magnetic Hysteresis Loops

Figure 3

4
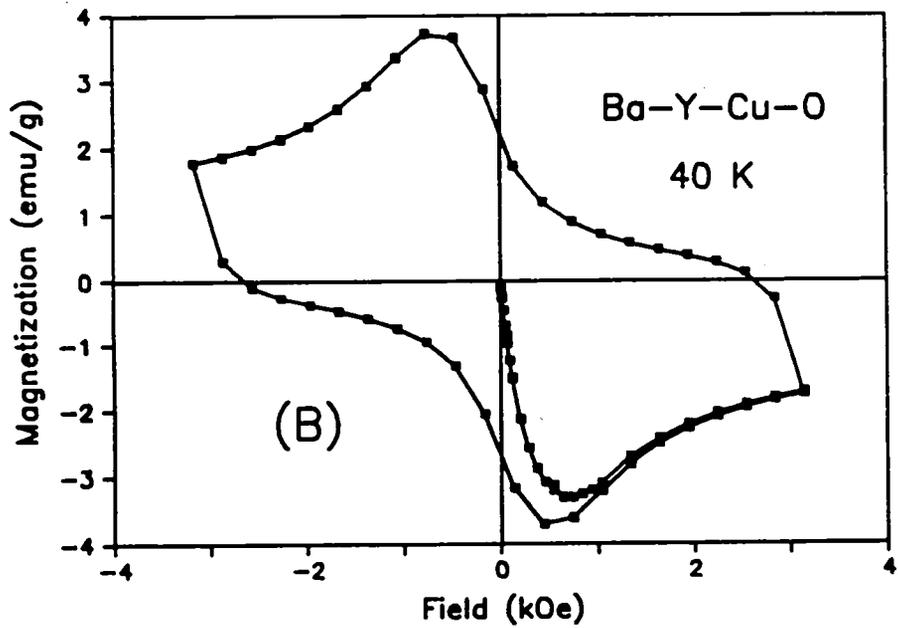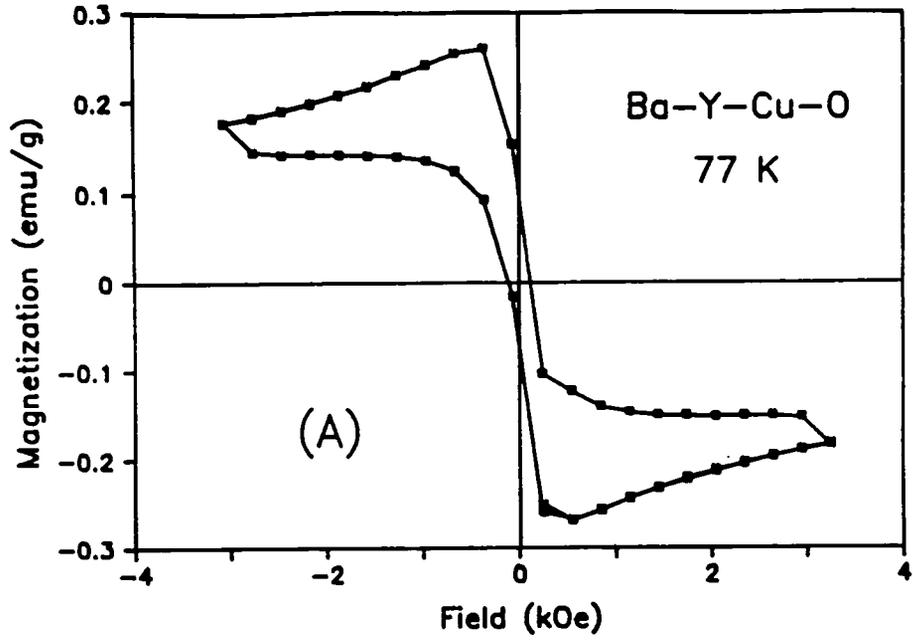
The $YBa_2Cu_3O_{7-x}$ is diamagnetic when $T<T_c$ (this is the definition of a superconductor). There is not much difference between the two experiments in Figure 2 performed on this material at 77 K. The difference in the levitating ability of the superconductor between field-cooling and zero-field-cooling the material is a reflection of the flux pinning of the material. At 77 K, Figure 3A also shows the lack of any significant flux trapping: the width of the loop is small, enclosing a small area, and the magnetization value on reducing the field to zero after applying 3 kOe is very small. In fact, for this material (density, ρ, of 6 g/cm^3) the trapped flux, which is equal to $4\pi M_r\rho$, is just 7 Oe. Since the samarium cobalt magnet possesses a field of over 2 kOe, the difference in levitation force would hardly be noticeable. However, at 40 K the trapped flux would be about 140 Oe and the difference between the Meissner effect and the magnetic shielding effect should be detectable.

The interesting possibility about an attractive force existing at 40 K in this material is true. For small fields (~140 Oe), the net susceptibility is positive, and therefore would act to pull the superconductor toward the magnetic field (i.e., the magnet). Of course, as the magnet approaches the superconductor, the field at the superconductor increases to the point where the $YBa_2Cu_3O_{7-x}$ susceptibility becomes negative and the force on the magnet becomes repulsive again.

When the magnet is pushed to the edge of the superconductor, the forces on it are no longer symmetrical; and since there is a repulsive force between the two objects, the magnet will be pushed outside the edge of the superconductor. When the magnet is placed on top of the partial disc, the edges are much closer and the region in which there is stable levitation becomes much smaller. In addition, due to the irregular topography, a smaller region of stability results from an increased localization of the induced currents in the partial superconductor.

FLUORESCENT PENETRANT INSPECTION

Dr. Sankar Sastri
New York City Technical College

OBJECTIVE: The purpose of this experiment is to familiarize the student with fluorescent penetrant inspection and to relate it to classification of various defects.

The penetrant method of nondestructive testing is a method for finding discontinuities open to surface in solids and essentially nonporous bodies. The method employs a penetrating liquid which is applied over the surface and enters the discontinuity or crack. After the excess of penetrant has been cleaned from the surface, the penetrant which exudes or is drawn back out of the crack indicates the presence and location of a discontinuity.

EQUIPMENT: Zyglo inspection equipment: ZL-15 fluorescent penetrant; ZP4 powder; black light: samples with defects.

PROCEDURE:

1. Inspect samples and parts to be free of any rust, scale, burrs, grease, oil, dirt, and other contaminants which would tend to give irrelevant indications.

2. Apply the penetrant by immersion of parts in the bath. Allow sufficient time for penetration into all discontinuities. Minimum penetrating time is as follows:

Nonferrous castings, forgings, weldments	-10 min.
Ferrous castings, forgings, weldments	-20 min.
Carbide tipped parts	-30 min.
Ceramics	- 1 min.
Plastics	- 5 min.
Glass	- 5 min.

3. Parts shall be washed in water immediately after time for full penetration has been allowed. The water temperature shall be maintained between 90 and 120° F.

4. Dry parts as thoroughly as possible by exposure to clean air. The drying temperature shall not exceed 165° F.

5. Dip the dried parts in the developing powder. Developing time is as follows: 15 minutes - Aluminum; 30 minutes - other parts.

6. Enter the black light booth. Allow 30 seconds for darkroom adaptation time. Examine the parts under black light. The intensity of black light shall be 100 foot candles minimum at a distance of 15' measured perpendicularly from the front of the light filter.

7. Separate the parts into categories of defects such as forging laps, grinding cracks, hot tear, shrinkage, porosity, and cold shuts.

REFERENCE: Nondestructive Inspection and Quality Control, ASM Metals Handbook, Vol. 11, 8th Edition.

SOURCE OF SUPPLIES: Magnaflux Corporation, Chicago, Illinois.

MAGNETIC PARTICLE INSPECTION

Dr. Sankar Sastri
New York City Technical College

OJBECTIVE: The purpose of this experiment is to familiarize the student with magnetic particle inspection and relate it to classification of various defects. Magnetic particle inspection is a method of detecting the presence of cracks, laps, tears, inclusions and similar discontinuities in ferromagnetic materials such as iron and steel. This method will most clearly show defects that are perpendicular to the magnetic field (Fig. 1).

The Magnaglo method uses a liquid containing fluorescent magnetic particles. The liquid is sprayed on the workpiece to be inspected and the part is magnetized at the same time. The workpiece is then viewed under black light and the presence of discontinuity is shown by the formation of a bright indication formed by the magnetic particles over the discontinuity.

EQUIPMENT: Y5 magnagflux yoke it, fluorescent magnetic penetrant, black light, samples with defects.

PROCEDURE:
1. Inspect samples and parts to be free of any rust, scale, burrs, grease, oil, dirt, and other contaminants which would tend to give irrelevant indications.

2. Place the Y5 yoke on the workpiece perpendicular to the direction of suspected cracks.

3. Trigger the switch and apply the inspection medium.

4. Inspect the part for any crack or defects. The yoke is capable of finding defects in a 25 square inch area across the magnetic field.

5. Locate the defect by circling with a red pencil and separate the parts into types of defects such as shrinkage porosity, gas porosity, cold shuts, and cracks.

6. Demagnetize the piece.

REFERENCE: Nondestructive Inspection and Quality Control, ASM Handbook, Vol. 11, 8th Edition.

SOURCE OF SUPPLIES: Magnaflux Corporation, Chicago, Illinois
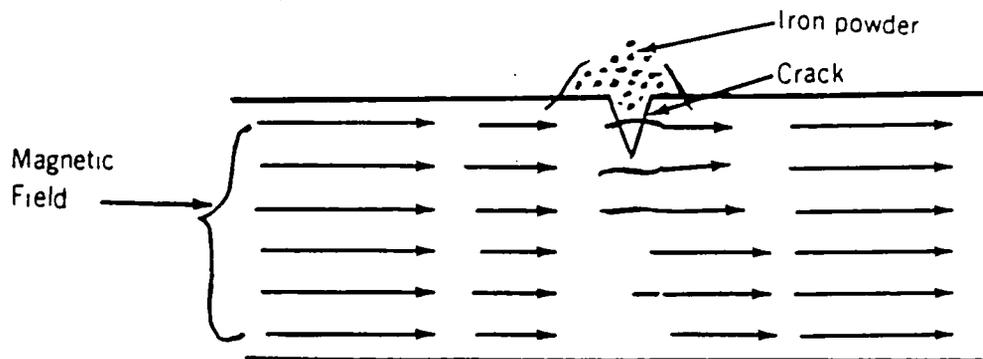
Principle of Operation
FIGURE 1

RADIOGRAPHIC INSPECTION

Dr. Sankar Sastri
New York City Technical College

OBJECTIVE: This experiment has several objectives which will familiarize the students with

1. Use of radiographic inspection system
2. The method of taking a radiograph
3. Dark room procedures in developing
4. Inspection of a radiograph

EQUIPMENT: Faxitron model 8075 radiographic inspection system

PROCEDURE:

1. Since X-rays are potentially dangerous, students should follow all safety procedures posted on the X-ray machine.

2. Turn the automanual selector switch to manual.

3. Adjust the KVP control to zero. Open the exposure compartment door and place the film on the shelf and the specimen in such a manner as to afford the best radiographic view. Place the identifying lead numbers in suitable places. Close the door completely.

4. Adjust the KVP control for the desired voltage on KVP meter.

5. Turn the timer knob to the desired exposure duration (for the stainless steel bracket casting) KVP <u>130</u>, <u>MA</u> 3, time <u>30</u> sec.

6. Press the expose button.

7. After set time, the expose light will extinguish. Adjust KVP control to zero. Remove the film.

8. Process the film by developing it for five minutes with agitation in developer. Rinse it in water before putting the film in fixer. After five minutes in fixer, wash the film in water for thirty minutes and dry it in the dryer for 30 minutes.

9. Inspect the radiograph in light carefully to see any defects such as porosity crack, shrinkage, hot tear, impurities, etc. Compare the defects with A.S.T.M. standards and locate the area in the actual piece.

REFERENCE: Nondestructive Inspection and Quality Control, ASM Handbook, 8th Edition.

SOURCE OF SUPPLIES: Picker International, Trevose, PA 19047

SAFETY FACTORS

RADIOGRAPHY

1. The radiation safety features of X-ray systems are designed, built, and tested to conform to the Exempt Protective Installation classification as defined in Section 3.1 of the National Bureau of Standards Handbook 93.

2. Use a radiation meter of the type and quality Victoreen 440 to check X-ray systems upon installation. Perform maintenance periodically.

3. Use a film badge service for cumulative individual monitoring.

ZYGLO

1. Flammability of penetrant materials
2. Volatility of penetrant materials
3. Annoyance created by dust of dry powder developers if not properly ventilated
4. Decomposition of chlorinated solvents at high temperature
5. Skin reaction – long or frequent exposures to penetrant materials

MAGNAFLUX

In addition to safety procedures of Zyglo, do not wear any watches that are not antimagnetic.

Demagnetize after inspection.

CONTACT ANGLE DETERMINATION PROCEDURE AND DETECTION OF AN INVISIBLE SURFACE FILM

G. Meyer and R. Grat
ramé-hart, inc.

INTRODUCTION, OBJECTIVES AND KEY WORDS

INTRODUCTION

The contact angle value, i.e., the tangent angle of a drop of liquid resting on a planar solid surface is a basic parameter which can be applied in a wide range of applications. One example of the successful application of this type of study was to create a cleaning process which permitted the salvage of much of the equipment which had been installed on an aircraft carrier which caught fire during construction, and which was damaged by smoke, oil and sea water contamination [1]. The work of developing suitable cleaning techniques was carried out at the Naval Research Laboratory under Dr. William A. Zisman who devoted a great deal of effort to the establishment of contact angle measurement as a practical tool for solving many problems concerning wetting interactions of liquids on surfaces.

The book <u>Contact Angle, Wettability and Adhesion</u> [1] did much to promote this science. Many other researchers, publications and short courses have greatly contributed to the understanding and utilization of this basically simple measurement technique, while providing a readily available source of information for others interested in applying it.

The purpose of this tutorial is not to provide highly technical information concerning contact angle theory, associated calculations and potential applications. (Such information is readily available from other sources.) The goal is rather to provide a basic understanding of the measurement technique, and to present a simple illustration that can be applied as a quality control method; namely, detection of a surface contaminant which exists on a surface that appears clean to the unaided eye.

References provided with this presentation should prove helpful to those wishing to explore other applications of contact angle data, or to gain knowledge of associated theory.

OBJECTIVES:

1. To acquaint the experimenter with the technique of measuring contact angles.

2. To conduct an experiment to illustrate how an invisible film on the surface of a substrate can be quickly detected using a relatively inexpensive instrument.

3. To create an awareness of contact angle measurement and provide references to assist those wishing to learn more about the topic.

KEY WORDS:

CONTACT ANGLE:
The tangent angle value of a sessile drop at its interface with a horizontal substrate. (Refer to CONTACT ANGLE, WETTABILITY AND ADHESION[1], Relation of the Equilibrium Contact Angle to Liquid and Solid Constitution, W.A. Zisman, for a discussion of this topic.)

This value is dependent upon the surface tension of the solid phase, the surface tension of the liquid phase and the surface tension of the vapor phase (normally air), surrounding the liquid/solid system.

SESSILE DROP:
A drop of liquid resting upon a horizontal substrate, known also as the "Liquid or Drop Phase".

SUBSTRATE:
A solid, planar surface, upon which the sessile drop is placed, known also as the "Solid Phase".

LIQUID/SOLID INTERFACE:
The common boundary shared by the liquid (sessile drop), and solid (Substrate Surface), phases of the experiment.

WETTING/SPREADING:
A liquid/solid system in which the liquid wets the surface of the solid has a resultant contact angle of zero. Contact angles having relatively low values indicate that the liquid is capable of spreading readily across the surface of the solid, without spontaneously wetting it. Contact angles of high values indicate that the liquid resists spreading.

PENDANT DROP:
A small drop extending from and still attached to the tip of a vertically oriented, drop-phase dispensing needle.

MICRO-SYRINGE ATTACHMENT:
An accessory to the contact angle goniometer, used to form the pendant drop of liquid and place it, in the form of a sessile drop, onto the substrate surface.

CONTACT ANGLE GONIOMETER:
An optical instrument employing a low-power, horizontally oriented microscope having a graduated reticule assembly, used to determine the contact angle of a liquid solid system.

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PREREQUISITE KNOWLEDGE REQUIRED

A general background in laboratory experimental procedures, including a basic understanding of microscopy.

EQUIPMENT AND SUPPLIES

Two standard glass microscope slides [a]

Butcher's Bowling Alley Paste Wax [b]

Distilled water, used to form the sessile drop [c]

Contact Angle Goniometer, ramé-hart, inc. Model 100-00(115), with Micro-Syringe Attachment Model 100-10 and 22 gage Drop-Forming Needle. [d]

EXPERIMENTAL PROCEDURE

Specimen Preparation:

Uncoated Specimen:

1. The slide was carefully removed from a freshly opened box of precleaned slides and five contact angle readings immediately taken at different locations on one of its surfaces, as described below.

2. The slide was handled only by its edges and extreme care was exercised at all times to prevent contamination of the test surface.

Coated Specimen:

1. This slide also was very carefully handled to prevent contamination.

2. A small amount of the wax was applied as evenly as possible to one surface of the slide, using a lens tissue and applying the wax with a circular motion.

3. The wax film was allowed to dry under ambient conditions for approximately 20 minutes.

4. The coated surface was gently buffed employing a clean, dry lens tissue and using a straight-line motion, parallel to the long axis of the glass slide.

Instrumentation:

NRL Contant Angle Goniometer, Model 100-00, [Fig. 1], is comprised of four major components:

Optical Bench approximately 500 mm long.

Horizontally mounted Goniometer/Microscope assembly containing a reticule assembly having two independently rotatable crosshairs, (known as the "base-line" and "measuring" crosshairs), and a graduated scale having one-degree divisions.

A stage assembly, equipped with screw-driven elevation and left-to-right motions, and a rack-and-pinion focusing motion. This stage is used to support and position the substrate during the experiment.

An illuminator assembly, equipped with a green filter, variable-intensity controller, and lamp-positioning/focusing controls.

Micro-Syringe Attachment, Model 100-10:

This drop-dispensing attachment is mounted to the stage of the goniometer and employs a micrometer-operated glass syringe assembly equipped with a Teflon piston. A 22-gage stainless steel, "square-ended" needle is affixed to the discharge end of the glass barrel and functions as the drop-forming tip.

The micro-syringe itself is mounted on a moveable head assembly which permits the needle-tip to be accurately pre-positioned above the substrate, readily raised from the area when not needed, and accurately repositioned to its original location when again needed.

Instrument Setup:

1. The contact angle goniometer is set up in accordance with its instruction manual. The micro-syringe is filled with distilled water and the micro-syringe installed in its mount.

2. Adjustments are made as required to position the needle tip on the optical axis of the instrument with the drop-forming needle tip approximately 3 mm above the substrate. The micro-syringe attachment's head assembly is then lifted and rotated to a raised standby position.

3. The illuminator's control is turned on and the variable intensity control set to produce a low level of light. The light level is gradually increased to a comfortable working level, while viewing through the microscope.

   CAUTION: To prevent eye injury, care must be taken to prevent setting of the brightness to an excessively high level.

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Goniometer Microscope - Alignment and Focusing:

NOTE: An inverted image is observed through the microscope due to the optical characteristics of the lens systems employed.

1. The microscope slide substrate is placed centrally on the goniometer's specimen stage top plate.

2. The elevation of the stage assembly is adjusted to position the top surface of the substrate approximately on the optical axis of the microscope.

3. The microscope is focused on the edge of the substrate located nearest the microscope, using the focusing knob located on the left side of the microscope base.

4. The stage elevation knob and baseline crosshair rotation adjustment are employed to align the top surface of the substrate with the baseline crosshair, defining what will become the liquid/solid interface area. [Fig. 2A]

5. The micro-syringe attachment's head assembly is now returned to its working position and the drop-forming needle tip enters the field of view.

Drop Formation and Placement:

1. The stage is now realigned as necessary and the microscope readjusted, bringing the drop-forming needle-tip sharply into focus.

2. A small pendant drop, approximately 1.5 mm diameter, is formed at the needle tip.

3. The micro-syringe is lowered using the screw adjustment provided, allowing the bottom of the pendant drop to contact the surface of the substrate.

4. The micro-syringe is again raised to the standby position, causing release of the pendant drop from the needle tip, forming the sessile drop upon the substrate surface.

5. The microscope is readjusted to produce a sharply focused image of the drop boundary. [Fig. 2C]

6. The illuminator's lamp position and focusing adjustments are set to produce a sharply defined silhouette image of the sessile drop.

Contact Angle Determination:

1. The measuring crosshair is adjusted to an angle which is somewhat less than the apparent contact angle, and the stage is readjusted to cause the measuring crosshair to become tangent to a point on the drop boundary which is located between the drop's base and crest. [Fig. 2D]

   This technique creates a visual aid which greatly lessens operator subjectivity in adjusting the measuring crosshair to its final position by providing a small wedge of light which can be used to accurately set the crosshair.

   The light wedge is bounded on one side by the measuring crosshair, on its second side by the drop boundary, and by the substrate surface on its third side.

2. The measuring crosshair is now rotated toward the base of the drop, and the stage moved left to right (or right to left), to maintain a tangent condition between the drop boundary and the measuring crosshair. As these adjustments are continued, the light wedge diminishes and finally ceases to exist. When this occurs, the measuring crosshair is tangent to the drop boundary at the liquid/solid interface, and the measuring crosshair is set to the contact angle value. [Fig. 2E]

3. The contact angle value is now read from the reticule assembly at the short vertical line located at the "Six O'Clock Position" of the reticule.

DATA ACQUIRED AND RESULTS:

Data

	Uncoated Specimen					Coated Specimen				
	#1	#2	#3	#4	#5	#1	#2	#3	#4	#5
Contact Angle, Degrees:	8.0	7.5	7.0	7.0	6.0	70.0	71.0	71.0	72.0	73.0
Mean Contact Angle:			7.1					71.4		

Results

The different surface tensions at the liquid/solid interfaces (clean glass on the uncoated substrate, a wax film on the other) have caused contact angles of distinctly different values to form.

The use of the contact angle goniometer has enabled the user to quickly ascertain the presence of a surface contaminant and to quantify the resultant contact angle value. Such information can be applied in a number of ways to improve formulations, optimize processes and function as a quality control method.

COMMENTS:

1. The same basic test could be employed to determine if a hand lotion intended to protect the skin resists wetting(and soiling), or conversely, if a formulation or process designed to remove soils from a surface actually accomplishes the task.

2. As localized surface effects may influence the contact angle value, several drops should be placed upon the substrate surface and their contact values determined. These values should be averaged to ascertain the mean contact angle value which should be employed in any subsequent calculations.

    CAUTION:

    Mid-Range contact angle values (20 to 70 degrees) can be readily measured with good precision: it is possible to estimate their value to 0.25 degrees. Contact angle values beyond these limits become more subjective. In general, the manufacturer of the rame-hart, inc. Model 100 Goniometer has indicated reliable repeatability of +/- 2 degrees; however, many users of this instrument have indicated their feeling that following their initial acclimation to the instrument, repeatability is in the area of +/- 0.5 degrees for mid-range contact angle values.

3. It should be noted that contact angle studies have been successfully employed in a tremendously wide range of applications. A few of these are

    Adhesion Studies: Adhesive and coatings formulation optimization.

    Surface treatment evaluation: research, development and quality control.

    Biomedical implant materials surface characterization.

    Printing ink formulation and printing roller surface studies.

    Oil reclamation: tar sands and tertiary oil recovery technique studies.

    Evaluation of the spreading effects of lubricants.

    Studies involving biological surfaces, e.g., stomach lining and human skin.

4. Although a wide variety of applications can be accommodated with the basic instrumentation demonstrated in this experiment, other applications require the use of temperature or atmospheric control, or the use of photographic attachments or other special accessories. A well-designed contact angle apparatus should be of modular construction, which permits the addition of such accessories. [Fig. 3].

CONCLUSION

The simple experiment presented has demonstrated that contact angle values can be readily determined and the resultant information applied in a useful manner. It has also demonstrated that the equipment is relatively simple and easily used. Our experience as a supplier of surface science instrumentation over a period of some twenty years indicates that

> Contact angle studies are readily carried out with relatively inexpensive equipment and produce meaningful data.

> Application of contact angle data frequently produces dramatic results in problem solving, formulation or process optimization and cost reduction.

> The existing reference base and educational programs available greatly assist in implementing contact angle-based research and development programs.

> Even in situations where textbook results are not experienced, the studies produce meaningful, problem-solving results.

SOURCES OF EQUIPMENT AND SUPPLIES EMPLOYED IN THE EXPERIMENT:

a.	Glass microscope slides:	Local Scientific Equipment Supply House
b.	Butcher's Bowling Alley Paste Wax:	The Butcher Polish Company Marlborough, MA 01752
c.	Distilled water	Local Scientific Equipment Supply House
d.	Contact Angle Goniometer and accessories:	ramé-hart, inc. 43 Bloomfield Avenue Mountain Lakes, NJ 07046

REFERENCES:

1. "Contact Angle, Wetability and Adhesion: Advances in Chemistry Series, Volume 43.

   Special Issue Sales
   American Chemical Society
   1155 16th Street, Northwest
   Washington, D.C.

2. "A Bibliography of Contact Angle Use in Surface Science"

   rame-hart, inc.
   43 Bloomfield Avenue
   Mountain Lakes, NJ 07046

Although the following publications are not referred to in the text of this presentation, they contain a great deal of information related to contact angle studies and related topics.

3. "Adsorption at Interfaces"
   K.L. Mittal
   A.C.S. Symposium Series No. 8, 1961
   A.C.S., Washington, DC

4. "Adsorption from Aqueous Solution"
   Advances in Chemistry Series, Volume 79, 1968
   A.C.S., Washington, DC

5. "Chemistry and Physics of Interfaces", 1965
   A.C.S., Washington, DC

6. "Chemistry of BioSurfaces", Volume 2, 1972
   M.L. Hair, Editor
   Marcel Dekker, New York, NY

7. "Dictionary of the Physical Sciences", 1987
   Cesare Emilliani, XXVI
   Oxford University Press

8. "Guidelines for Physicochemical Characterization of Biomaterials"
   U.S. Department of Health and Human Services
   National Institutes of Health Publications No. 80-2186, September, 1980

9. "Interaction of Liquids at Solid Substrates"
   Advances in Chemistry Series, Volume 87, 1968
   A.C.S., Washington, DC

10. "Physical Chemistry of Adhesion", 1971
    D.H. Kaelble
    John Wiley and Sons, Inc., New York, NY

11. "Physical Chemistry of Surfaces", Third Edition
    Arthur W. Anderson
    Wiley Interscience, New York, NY

12. "Solid Surfaces and the Gas-Solid Interface:
    Advances in Chemistry Series, Volume 33, 1961
    A.C.S., Washington, DC

13. "Surface and Colloid Science", Volume 2, 1979
    R.J. Good and R.R. Stromberg, Editors
    Plenum Press, New York, NY

14. "Surface Chemistry: Theory and Industrial Applications", 1962
    Lloyd I. Osipow
    Reinhold Publishing Co., New York, NY

15. "Treatise on Adhesion and Adhesives", Volume I, 1967
    R.L. Patrick, Editor
    Marcel Dekker, New York, NY

NRL CONTACT ANGLE GONIOMETER
MODEL 100-00

FIGURE I

Specimen Stage

Substrate

Baseline Crosshair

Measuring Crosshair

A
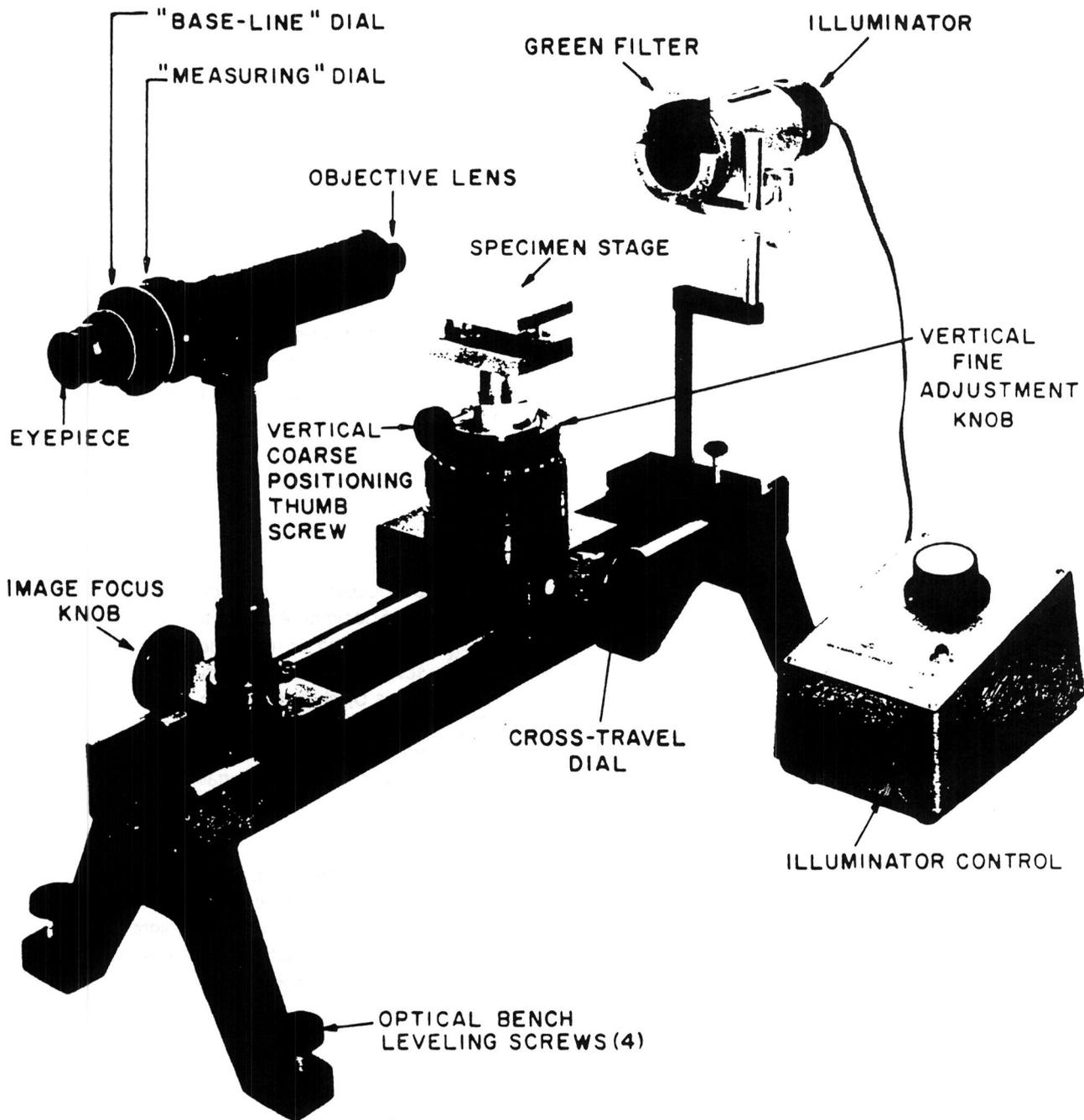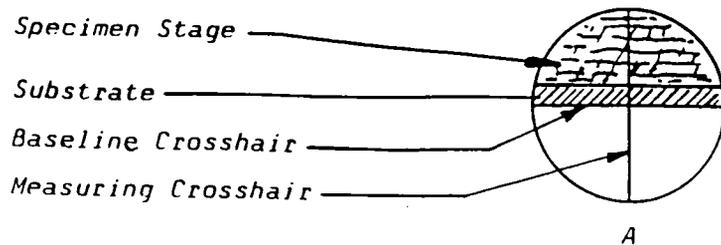
1. Focus on the top surface of the substrate at the edge nearest the Microscope. Adjust the substrate and the "base-line" to achieve coincidence. NOTE: Do not disturb this setting during the course of acquiring the reading.

Sessile Drop

Air

B
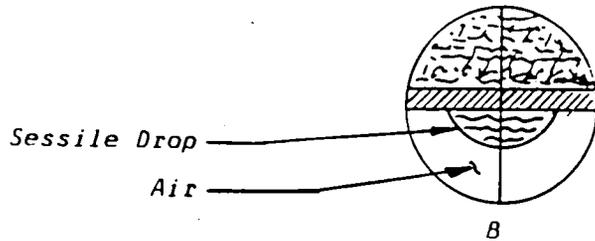
2. Deposit a small volume of liquid onto the substrate to form a sessile drop having a diameter of approximately 2.5 mm. The drop size may vary to suit certain studies.

C
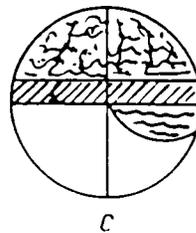
3. Refocus the Microscope and adjust the Specimen Platform to view the extreme left or right side of the sessile drop. Verify accuracy of focus by shifting the line of sight. If the drop profile appears to move relative to the "measuring" cross-line, proper focus is not attained.

D
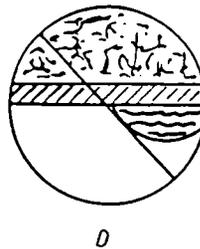
4. Adjust the "measuring" cross-line to tangency above the base of the drop to create a wedge of light bounded by the two cross-lines and the drop profile.

E
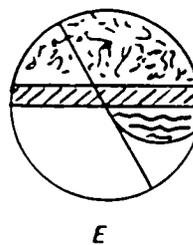
5. Slowly rotate the "measuring" cross-line while adjusting the cross-travel of the Specimen Stage Assembly so that the wedge of light is gradually extinguished and the cross-line attains tangency with the drop profile at the base of the drop.

Microscope Slide for Platform Leveling

FIGURE 2 .

24

TABLE 2
ALTERNATE OTPICAL COMBINATIONS

The following are examples of Microscope
Objective Lenses which may be substituted
for the Objective lens supplied with the
Goniometer. The user may acquire alternate
lenses from the manufacturers noted or from
others not listed. This table is intended
as a guide to assist the user in determining
the working distance which may be expected if
the overall system magnification is changed.

The overall system magnification is obtained
by multiplying the Objective Lens magnification
by 10 X (Eyepiece magnification). For users
who may desire to determine other optical
characteristics through calculations, the
optical tube length is 160 mm.

Objective Lenses available from Gaertner
Scientific Corporation, 1201 Wrightwood Ave.,
Chicago, IL 60614

mag.	Working dist.	cat#
1.0 ×	155m	M-226
1.4 ×	109mm	M-225-C
1.7 ×	89mm	M-225
3.2 ×	45mm	M-222
4.0 ×	35mm	M-220

Objective Lenses available from
The Ealing Corporation
22 Pleasant Street
South Natick, MA 01760

mag.	Working dist.	cat#
1.0 ×	150m	11-8216
1.5 ×	100mm	11-8224
2.0 ×	75mm	11-8232
4.0 ×	35mm	11-8240
6.0 ×	20mm	11-8257
10.0 ×	15mm	11-8265

Diagrams (1–17):

1. AIR / SOLID ⟨A⟩
2. GAS / SOLID ◇
3. VAPOR / SOLID ◇
4. VACUUM / SOLID ◇
5. GAS LIGHT LIQUID / HEAVY LIQUID ◇
6. VAPOR LIGHT LIQUID / HEAVY LIQUID ◇
7. VACUUM / HEAVY LIQUID ◇
8. LIGHT LIQ. HEAVY LIQ. / SOLID ◇ OR ◇
9. LIGHT LIQ. MEDIUM LIQ. / HEAVY LIQUID ◇ OR ◇
10. SOLID / LIGHT LIQ. HEAVY LIQUID ◇ OR ◇
11. SOLID / GAS BUBBLE LIQUID ◇
12. AIR / HOT MELT OR LIQUID ⟨A⟩
13. GAS / HOT MELT ◇
14. VAPOR / HOT MELT ◇
15. VACUUM / HOT MELT ◇
16. LIGHT LIQ. / HEAVY LIQUID ⟨B⟩ OR ◇
17. HEAVY LIQ. / LIGHT LIQUID ⟨B⟩ OR ◇
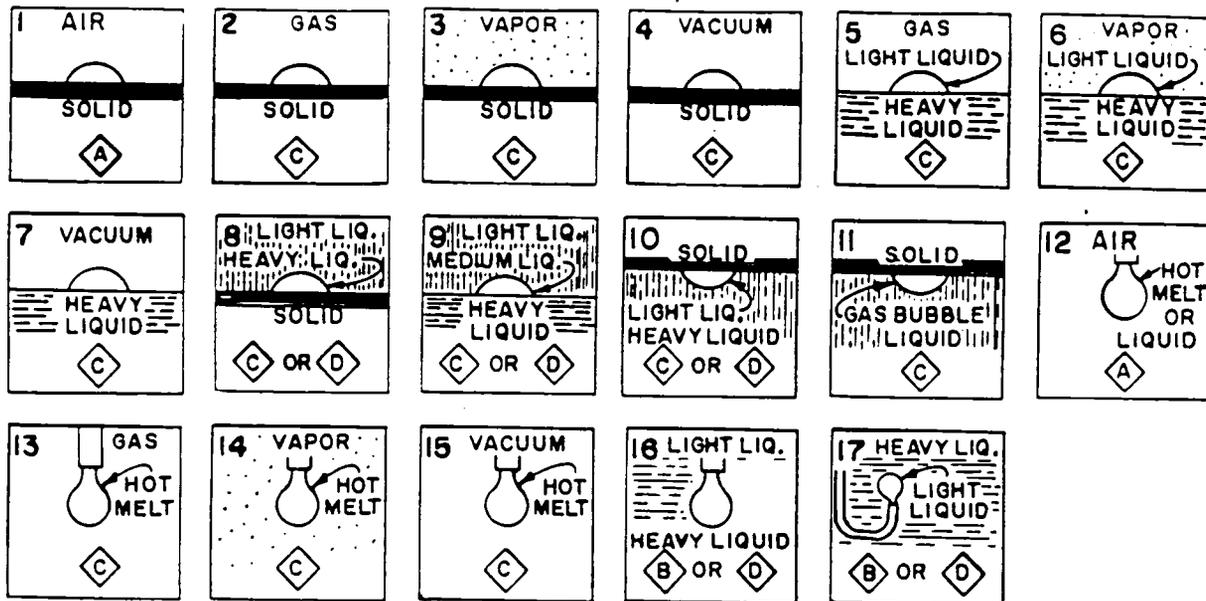
X = REQUIRED

O = OPTIONAL

◇ = REQUIRED FOR ENVIRONMENT NOTED

"GAS" = INERT GAS

Ⓐ = AMBIENT ATMOSPHERE & TEMPERATURE

Ⓑ = TEMPERATURE TO 300°C. ONLY

Ⓒ = TO 300°C., VACUUM, & TO 10 PSIG

Ⓓ = TO 230°C. & TO 1000 PSIG

Ⓔ = HOT MELT PENDANT DROP - TO 230°C.

INSTRUMENT OR ACCESSORY	CATALOG Nº		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	
NRL CONTACT ANGLE GONIOMETER	100-00	A	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	
ENVIRONMENTAL CHAMBER	100-07	C		X	X	X	X	X	X	Ⓒ	Ⓒ	Ⓒ	X		X	X	X	Ⓑ	Ⓑ	
CHAMBER COVER WITH STAGE	100-09-10	C		O	O	O				O		Ⓒ	X			X	X			
HIGH PRESSURE CHAMBER	100-08/30	C								Ⓓ	Ⓓ	Ⓓ						Ⓓ	Ⓓ	
" " -INVERTED NEEDLE	100-08-17	–																	Ⓓ	
MICRO-SYRINGE ATTACHMENT	100-10	–	X	X	X	X	X	X	X	Ⓒ	Ⓒ	Ⓒ	X	Ⓐ				Ⓑ	Ⓑ	
" " -INVERTED NEEDLE	100-10-13	–										Ⓒ	X						Ⓑ	
ELEVATED TEMP. SYRINGE	100-11	E	O	O	O	O								Ⓔ	X	X	X	Ⓑ		
FILM CLAMPS	100-15	A	O																	
IMMERSIBLE FILM STAGE	100-16	C		O	O	O				O										
ROTATING IMMERSIBLE STAGE	100-18	B	O							O		O	O							
TILTING BASE	100-06	FOR ADVANCING & RECEDING CONTACT ANGLES																	
35mm CAMERA	100-13	FOR RECORD/MEASUREMENT PHOTOGRAPHS																	
POLAROID CAMERA	100-14	" " " "																	
FINGER SUPPORT	100-19	FOR CONTACT ANGLES ON SKIN																	

NRL CONTACT ANGLE GONIOMETER
SYSTEM GUIDE

FIGURE 3

SENSITIZATION OF STAINLESS STEEL

Professor James P. Nagy

Erie Community College
Main and Youngs Road
Buffalo, NY

PREREQUISITE   The completion of the study of stainless steels in a basic course in
materials science.

OBJECTIVE   1. The objective of this experiment is to determine the corrosion
rates of 18-8 stainless steels that have been sensitized at
various temperatures.

2. Show the application of phase diagrams.

EQUIPMENT   Heat treat furnace
Analytical scale
1000 ML Polyethylene beaker
Polyethylene graduated cylinder
Metallurgical polishing equipment
Metallurgical microscope
Safety goggles, rubber gloves, and apron

SUPPLIES   304 stainless steel samples 1" X ½" X .032"
Cold mounting compound
Nitric acid
Hydrofluoric acid

PROCEDURE   The laboratory instructor will assign each student one of the
following temperatures to which the sample will be heated.
Identify the samples as follows:

TEMPERATURE	IDENTIFICATION
As received	grind off one corner
550 C	grind off two corners
600 C	grind off three corners
650 C	grind off four corners
700 C	grind off no corners
750 C	bend one corner
800 C	bend two corners
850 C	bend three corners
900 C	bend four corners
950 C	cut one notch in sample
1000 C	cut two notches in sample
1050 C	cut three notches in sample

1. Bend the sample so that it is "U" shaped.

2. Heat the sample to the assigned temperature for one half hour and
then water quench the sample.

3. Weigh the sample and record the weight.

4. The laboratory instructor will prepare the following test solution in a 1000 ML polyethylene beaker:

Hydrofluoric acid	15 ML
Nitric acid	50 ML
Water	435 ML

SAFETY NOTE: When making this test solution always add the acids to the water. Hydrofluoric acid will attack glass; polyethylene beakers and graduated cylinders must be used. This test solution will work at room temperature. When handling acids or test solution, always wear safety goggles, rubber gloves, and rubber apron.

5. After all the students in the laboratory class have heat treated and weighed samples, place all the samples in the test solution at the same time. It will take approximately 24—48 hours for the samples to corrode. If in that time the test solution turns a dark blue-green color, the solution is depleted. If more corrosion is desired, discard the solution and prepare a fresh one, and continue corroding the samples.

6. Weigh the sample; record the weight.

7. Mount the "U" shaped sample on its edge in a cold mounting material so the cross section of the sample can be observed and then polish the sample. Observe the edge of the sample under a microscope using low magnification.

REQUIRED

1. Determine the percent weight loss for all the samples.

2. Plot a graph of percent weight loss versus temperature.

3. Draw the sample showing the most intergranular corrosion.

4. Draw the sample showing the least intergranular corrosion.

QUESTIONS

1. Which of the samples corroded the most?

2. What was the reason the as-received and low temperature samples had little, if any intergranular corrosion?

3. Using a phase diagram, explain why the high temperature samples had little, if any intergranular corrosion.

4. What alloy addition could be added to this steel to retard intergranular corrosion? Explain why.

NOTES FOR THE LABORATORY INSTRUCTOR

This experiment has worked out very well for us. If your furnace will not go up to 1050 C, no problem. We can only get up to 900 C and that is good enough. If you can reach 1050 C, that would be better.

The samples are marked by cutting off corners or bending corners because the test solution will remove any markings put on the samples.

It will take about 24—48 hours to corrode the samples; you can tell when you have gone far enough. The samples heated to about 650—700 C will feel very granular on the surface. There are times when we have let the samples stay too long and the samples have completely dissolved!

ANSWERS TO THE QUESTIONS

1. The samples heated to 650 C—700 C will corrode the most because the stainless steel is sensitized the most in this temperature range.

2. The as-received sample and the ones heated to a low temperature were not sensitized because chromium carbides were not formed at the grain boundaries.

3. The samples heated to high temperatures were in the all gamma region of the phase diagram, and upon quenching, the carbon was held in solution.

4. In stainless steels, the alloys of Ti, Nb, and Ta will tie up the carbon so that no chromium carbides will form at the grain boundaries.

REFERENCE

Brick, Pense and Gordon, Structure and Properties of Engineering Materials. Fourth Edition, New York: McGraw Hill, P. 340.

SENSITIZATION OF STAINLESS STEEL

DATA SHEET

TEMPERATURE OF SAMPLE	WEIGHT BEFORE TEST	WEIGHT AFTER TEST	PERCENT WEIGHT LOSS	COMMENTS ON THE MICROSTRUCTURE OF THE SAMPLE
As Received				
550°C				
600°C				
650°C				
700°C				
750°C				
800°C				
850°C				
800°C				
950°C				
1000°C				
1050°C				

Fe. 18Cr, 8Ni   wt. % C

THE USE OF COMPUTERS IN A MATERIALS SCIENCE LABORATORY

by J. P. Neville, Prof. Material Science,
Wentworth Institute of Technology, Boston.

PREREQUISITE KNOWLEDGE REQUIRED FOR EXPERIMENT: None

OBJECTIVE: To make available a method of easily recording the microstructure of a sample by means of a computer. This method requires a minimum investment and little or no instruction on the operation of a computer.

EQUIPMENT AND SUPPLIES: The following is one set of commercially available equipment which will easily capture microstructures at a low cost and also be useful as a regular computer.

An **RCA** model TC 2511 camera with a 16 mm lens was used to record the structures shown in this experiment. This camera was positioned in direct contact with a **Bausch & Lomb** metallurgical microscope, equipped with a 40X objective lens and a 5X eye piece. The image was fed into a **MacVision** unit, made by **Koala Technologies Corp**. The signal was then fed into a 512K **Macintosh** computer and the results printed out on the standard **Apple Imagewriter** dot matrix printer. The entire system costs less than $3000.

The choice of **Apple** equipment was influenced by both the low price and ease of operation. **Macintosh** has the added feature of allowing the microstructures to be saved as a "MacPaint" document. In this program the structure is easily modified, text can be added, or features can be enhanced. The larger capacity 512K machine was chosen because of its speed when programmed with an internal "Ram Disc". The operation is almost instantaneous in this mode and the added cost over the 128K model is minor. An SE model could also be used.

PROCEDURE: The setup needed to view microstructures is as simple as placing the lens of the TV camera in direct contact with the eye piece of the microscope. For ease of focus, a metallurgical microscope which has two viewing lenses is ideal. This permits adjusting the field of view without removing the camera and directly comparing the original with the structure projected on the computer screen. The student has control over the brightness and contrast of the pictures. The pictures obtained can be lightened to only show outlines, with the idea of having the student fill in the details; or the pictures can be reproduced with various shades, capturing most of the phases present. Programs and equipment have been available for some time to analyze metallurgical microstructures but all have been expensive and tend to give more information than is needed by a student at the introductory level. The setup described here can be used to make good reproductions of microstructures in the lab or pictures of actual test equipment.

The pictures obtained do not contain all the fine details that are seen in the microscope or with a camera. For example, the fine grain boundaries present in some microstructures are not reproduced by inexpensive systems. However, this is both an advantage and a disadvantage to the student who is exposed to microstructures for the first time. While the expert may well be frustrated by the missing details, the beginning student will easily be able to see gross differences in structure and learn how these changes affect

the behavior of the material. By only seeing the major contrasts of the sample, the student is not confused or overwhelmed by too much detail. Note that the differences in structure of white, malleable and nodular cast irons are easily identified. Distortion of grains due to cold working is also well defined.

Detail is best seen when the grains are different colors. This may be brought out more by using a contrasting or "tint" etch which will color one phase more than another. **Metal Progress** Magazine, published by **ASM**, ran an article on tint etches in the Mar 85 issue, pages 31-41.

When the student advances in experience, it will be easy for him to add any missing features he saw in the microscope to his finished print. The structure can be worked on or improved by hand or by using computer graphics or drawing programs. Grain boundaries can be brought out and phases can be labeled using this technique, and the results can be included in a report.

Even without improving them, the pictures obtained are better than most students are capable of reproducing by hand, as seen below.
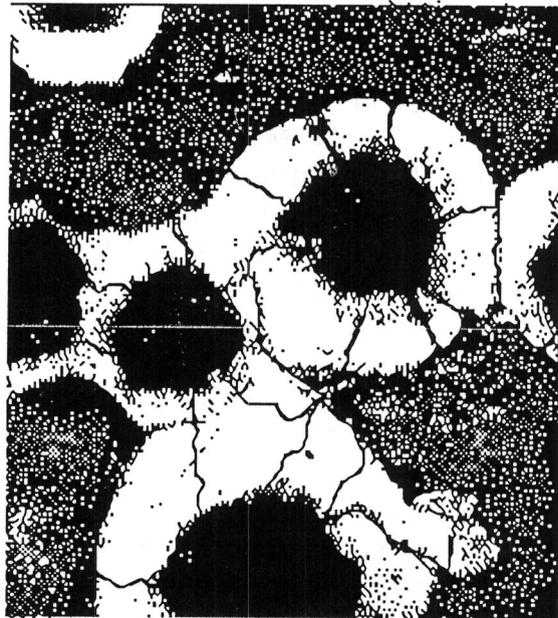
Original figure not available

REFERENCE: Neville, "The Computer and the Microscope," **Journal of Engineering Technology**, Vol 2, No. 2, p. 18-19.

DIAGRAM OF EQUIPMENT

Black & White TV Camera

BRIGHTNESS

CONTRAST

Digitizer control box

Macintosh™ computer

Metallurgical microscope

To the Printer

Irregular carbon

White Cementite
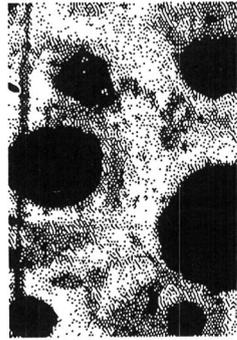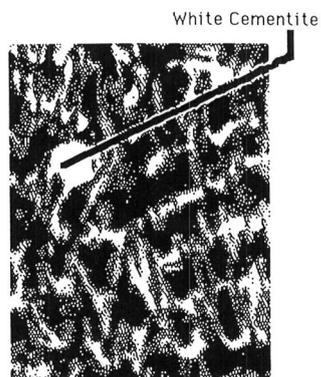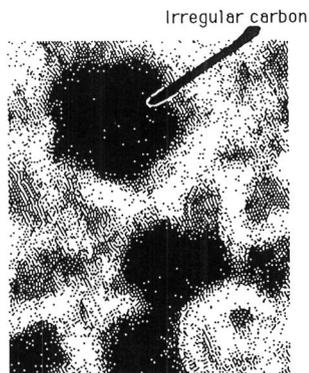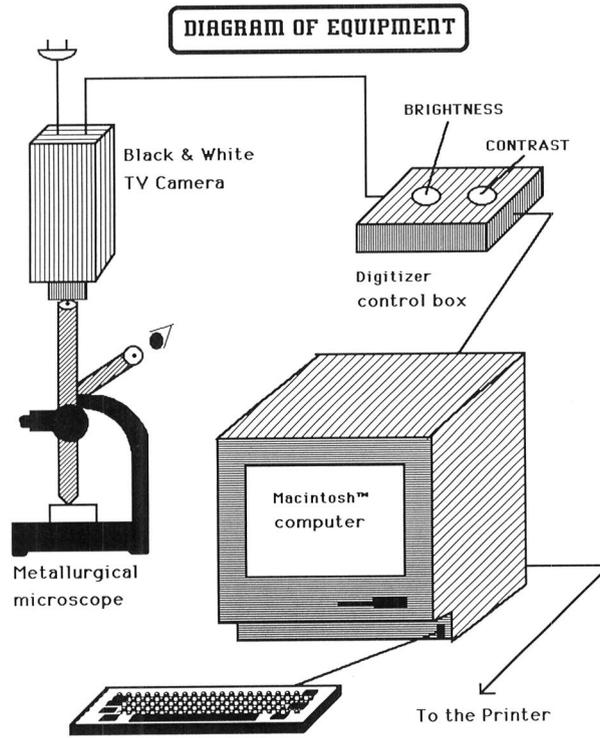
Flakes of Carbon

Round spheres of carbon

TYPICAL MICROSTRUCTURES CAPTURED BY
THE SET-UP DESCRIBED HERE

CRYSTAL GROWING
J. P. Neville
PREREQUISITE KNOWLEDGE REQUIRED FOR EXPERIMENT: NONE

OBJECT: To demonstrate the way crystals grow and how they affect the behavior of
material.

To compare the growth of crystals in metals and nonmetals.

EQUIPMENT:

Hot plates, glass plates, beakers, and glass rods; balance to weigh out the salt and
water; furnace crucibles, sand, safety glasses gloves, firebricks, metal tray, and
tongs. Materials in the form of Epsom salts, water, 50% Aluminum - Copper alloy
(previously formulated).

SAFETY:

Whenever you are dealing with hot liquids, there is the potential for burns and spills.
Protect yourself from the "possible", especially around the hot metal. Make sure
you know where any potential spillage will go and place something in the way to
protect yourself. In part B be careful of the hot metal. Remember, it will still be
very hot, even when it is a solid.

PROCEDURE:

PART A: A supersaturated solution of salt will separate into crystals on cooling.
The size of the crystals is a function of the rate of cooling, the amount of
impurities present and the degree of supersaturation (concentration of salt present).

1. Dissolve 25 grams of Epson salts in 25 ml of water. Heat the water
until all the salt dissolves, but keep below the boiling point. If all
the salt will not dissolve, add water in small quantities until it does.
Pour some of the solution onto a clean glass plate so as to form a thin
film and watch it solidify. If it solidifies too rapidly, return it to
your beaker, add some more water, and try again. If no crystals form,
return the liquid to the beaker and add more salt. When you achieve
the proper mixture, you should be able to observe the crystals start
to form at the edge and slowly "grow" toward the center.

2. Record the ratio of salt/water you used and sketch the crystals you saw,
giving particular attention to the area where two crystals meet or join.

PART B: If an alloy of two metals forms a eutectic and a solid solution (or
intermetallic compound) on cooling, the eutectic can be poured off and crystals
formed by the solid solution will be exposed.
This part of the experiment lends itself best to a demonstration. (Let the
instructor burn himself instead of you.) An alloy has been prepared by
melting equal weights of pure copper and aluminum together overnight in the
furnace. An intermetallic compound, $CuAl_2$, and a eutectic are formed. The
compound takes the form of large needles if slow-cooled.

1. Heat the copper-aluminum alloy up to approximately 700°C and pour it
into a hollowed out firebrick resting on a metal tray full of sand or a
hollow made in dry sand contained in a metal tray. USE CARE DURING THIS
TIME!

2. As the liquid cools you will first observe small bubbles on the surface
as the solubility of the dissolved gases decreases. Next, you may be

able to see a hint of motion just under the surface as the crystals of metal are forming. Tilt the pan slightly to see this structure. When there seems to be some rigidity or structure inside the liquid, tip the pan to let liquid (or eutectic) flow out. Depending on when you decide to tip the pan, you will see large crystals that have formed throughout the system. Note the size of the crystals in the liquid that was poured out as compared to those left behind.

3. Repeat the procedure again by remelting the mixture. Note how brittle this mixture is - most unlike copper or aluminum - Why? Sketch the shape of the crystals both in the hollow structure and those in the material that was poured off.

CONCLUSIONS:

Can you give some general conclusions as to why crystals grow the way they do? What can you conclude by comparing the growth of the metallic crystals as compared to the Epsom salts? What would you recommend doing if you wanted small crystals or if you wanted large crystals present in a sample?

REFERENCE: Neville, "Demonstration of Crystal Structure," Engineering Education, Vol. 75, No. 5 pp. 299-300.

A DEMONSTRATION OF CHILL BLOCK MELT SPINNING OF METAL

Robert B. Pond

Department of Materials Science and Engineering
Johns Hopkins University

Prerequisite knowledge

One of the most exciting adventures in materials in recent times has been the discovery of amorphous metals and the pursuit of methods of manufacturing various alloys into various shapes which are amorphous. Some of these alloys possess electrical properties which are extremely beneficial, whereas others offer different benefits such as corrosion resistance and no solidification shrinkage anomalies. There are a number of techniques for producing such amorphous shapes, but one of the earliest systems used is now referred to as CHILL BLOCK MELT SPINNING.

Objective, Equipment, and Supplies

A chill block melt spinning demonstration is a spectacular and intriguing exercise. The object of this demonstration is to show the simplicity of the process. The apparatus used in the demonstration can be quite simple, easy to construct, and extremely inexpensive.

The basic components of the apparatus consist of A) an ejection tube, B) a pressure bulb and hose to generate an ejecting force on the metal in the ejecting tube, C) a rotating chill block, and D) a drive motor for the chill block.

A and B) Since it is advantageous (for safety reasons) to use low melting temperature metals for this demonstration, a Pyrex glass tube works well as an ejection tube. Any tube size can be used, but a 10—12 mm size is convenient. One end of this tube should be sealed by heating and drawing the glass and the heated mass, then redrawn to produce a conical cavity with a small apex angle as illustrated in Fig. 1. The glass wall is then ground away to produce an orifice approximately 300—500 microns in diameter. See Fig. 2. The tube is cut to a length of about 25 cm and provided with a rubber hose which connects it directly to a hand operated pressure bulb.

A tin rod (having a diameter slightly less than the I.D. of the tube) when inserted and melted in the tube can now be ejected in a continuous stream by applying pressure on the squeeze bulb. If the free fall of the stream is sufficiently great to allow solidification before impingement on a solid surface, and if the stream velocity is sufficiently great to cause the Rayleigh break-up to be beyond the point of solidification, then a round tin wire will be produced.

C and D) A simple, but most effective chill block can be constructed from a new aluminum 9-inch cake pan. The exterior rim of this pan should be buffed to a high sheen. A new pan is generally delivered in this condition. The center of the pan can be located by balancing it on the point of a pencil. A small (6 to 8 mm diameter) hole is drilled in the center of the pan. A small bolt inserted into the hole provides an axle for the pan and allows it to be mounted on a hand drill. See Fig. 3.

Procedure

If the stream of molten tin previously described is directed at the rotating outer flat portion of the cake pan rim, then the circular cross section molten stream will be converted into a flat solid ribbon, the thickness and continuity of which will be dependent on the drill speed.

A standard hand drill will run at about 1000 revolutions per minute, and the rim surface of the cake pan will be traveling at approximately 1200 cm per second. The ribbon is therefore cast at a speed of 1200 cm per second. This is by no means the casting speed limit! Substitution of a higher speed motor will result in thinner fibers, higher quench rates, and greater casting speeds.

Although the dwell time (or distance) of the tin on the wheel is very small, it will tend to lengthen if the wheel becomes warm by extended operation.

It will be noticed that by controlling the point of impingement of the molten stream, one also controls the emanating trajectory of the solid fiber. Best results are obtained by using small attack angles (approximately 30 degrees) and molten stream flight distances of about 20 to 50 mm.

38

FIGURE 1

FIGURE 2

Cake Pan

Nut   Nut

Pressure bulb

ejection tube

Cake Pan

Hand drill

Ribbon Fiber.
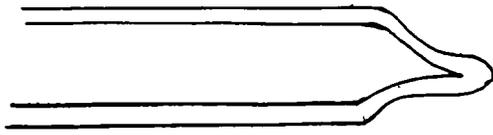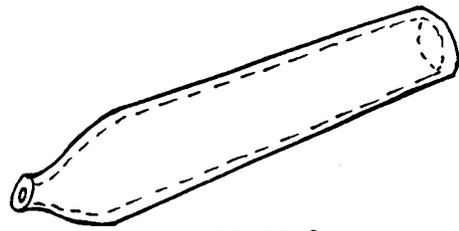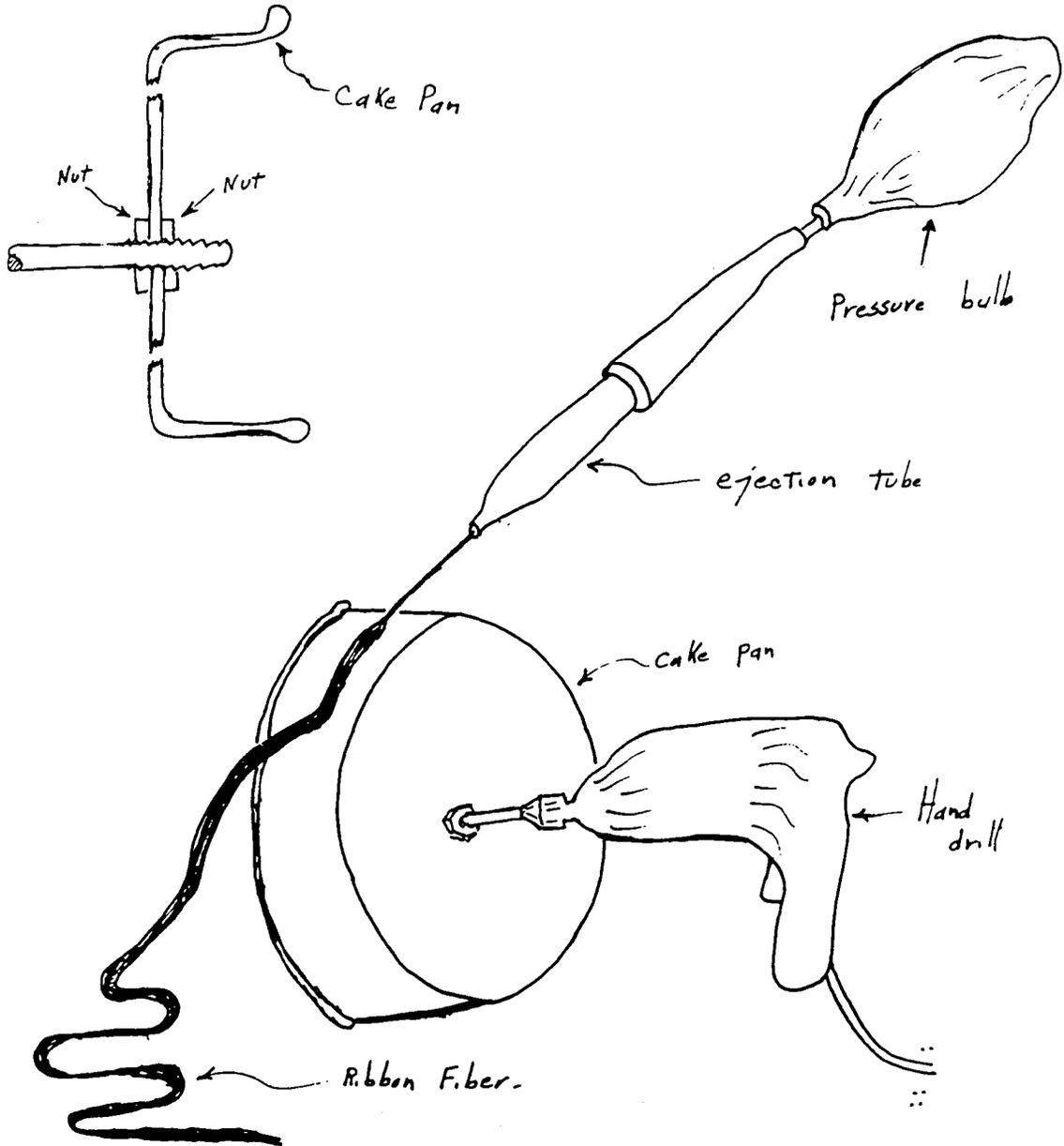
FIGURE 3

FRACTURE OF BRITTLE SOLIDS

R.D. Doherty and S.K. Nash
Department of Materials Engineering
Drexel University
Philadelphia, Pennsylvania

PREREQUISITE KNOWLEDGE

This experiment is one of four devoted to the mechanical properties of metals, polymers, and ceramics, as part of a one quarter laboratory course taken by sophomore materials engineering students. In addition to freshman calculus, chemistry, and physics, an intensive course in fundamentals of materials must precede or accompany the laboratory exercises.

OBJECTIVES

Upon completion of this experiment, the student should be able to 1) give an elementary account, orally or in writing, of brittle versus ductile failure, both from a theoretical and applications point of view, 2) conduct a three-point bend test on a tensile testing machine, and 3) statistically analyze experimental data that exhibit inherently wide scatter.

PROCEDURE

Attach a low capacity load cell (say 50 lbs.) to the tensile testing machine; calibrate the machine with known weights; attach a three-point bending jig (Figure 1), and adjust the machine to produce full-scale deflection on the chart recorder for a load of 10 lbs. While wearing latex gloves, open a fresh package of clear, 1 X 3 in. glass microscope slides. Stress five slides to fracture and record data. Repeat this procedure on slides that have been scored lengthwise and widthwise, respectively, on 400 grit silicon carbide abrasive paper. Figure 2 shows a simple holding fixture that helps produce uniform scratches. Next, repeat each of the aforementioned test conditions, but in addition, immerse each specimen in 5% HF solution for three to five minutes. (Tensile testing and immersion can be coordinated to save time.) When the tests have been completed, calculate the maximum (tensile) bending stress for each specimen in accordance with the beam formula shown under Figure 1. For each group of five fracture stress values, compute an arithmatic mean, standard deviation, and standard error of the mean. The report on this experiment includes a summary of relevant brittle fracture theory and all pertinent observations of specimen behavior in relation to the numerical data and the applicable theory.

CAUTION: a) Wear safety goggles throughout this experiment. b) Place a transparent (Plexiglas) screen around the test apparatus. c) When handling slides, wear latex gloves and hold a slide at its edges. Use a polypropylene beaker and Teflon-coated stainless tongs when immersing slides in HF solution; rinse slides in distilled water and dry in blast of warm air before testing. d) Always wear latex gloves when handling HF acid or its solutions.

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SOURCES OF SUPPLIES

Precleaned plain glass microslides, hydrofluoric acid (52%), Teflon-coated stainless steel tongs, and a polypropylene beaker are generally available in the chemistry stockroom; if not, enough of these expendable and reusable items for a large number of experiments may be purchased from a chemical supply house, e.g., Fisher or Thomas, for about $50.

TYPICAL EXPERIMENTAL RESULTS:
BRITTLE FRACTURE OF GLASS

CONDITION 1 - AS-RECEIVED GLASS SLIDES:

10 specimens
Breaking loads: 2.9, 5.7, 5.1, 5.5, 3.8, 5.1, 3.3, 5.9, 4.7, 3.5 kgs
Deflections: (last 5 readings)   0.9, 0.8, 1.1, 0.8, 0.8 mm
Mean fracture stress: **130 MPa**
Standard deviation: **-30 MPa**
Standard error of mean: (S.D./√n)   **-10 MPa**

CONDITION 2 - AS-RECEIVED GLASS SLIDES
AFTER ETCHING:

5 Specimens
Breaking loads: 20.1, 35.7, 30.7, 20.1, 17.7 kgs
Deflections: 1.8, 2.5, 2.6, 1.3, 1.6 mm
Mean fracture stress: **244 MPa**
Standard deviation: **-69 MPa**
Standard error of mean: (S.D./√n)   **-31 MPa**

CONDITION 3 - AS-RECEIVED GLASS SLIDES
AFTER ABRADING

5 Specimens
Breaking loads: 4.4, 5.0, 4.4, 4.3, 3.1 kgs
Deflections: 0.4, 0.4, 0.4, 0.4, 0.4 mm
Mean fracture stress: **42 MPa**
Standard deviation: **-6 MPa**
Standard error of mean: (S.D./√n)   **-3 MPa**

CONDITION 4 - AS-RECEIVED GLASS SLIDES
AFTER ABRADING AND ETCHING

5 Specimens
Breaking loads: 10.0, 8.9, 27.2, 29.0, 9.9 kgs
Deflections: 0.8, 0.8, 2.0, 2.2, 0.8 mm
Mean fracture stress: **165 MPa**
Standard deviation: **-90 MPa**
Standard error of mean: (S.D./√n)   **-40 MPa**

VISUAL OBSERVATIONS

1. Etching did not significantly affect the appearance of the slides. The abraded slides showed no detectable change, with the abrasion marks still visible. The as-received slides showed, after etching, some surface flaws. The etching has blunted the sharp cracks, but in so doing, has apparently made the flaws sufficiently wide at the surface to scatter light so that they could then be seen.

2. The strongest slides, those which after etching had fracture loads exceeding 20 kgs, were **very highly bent** just before fracture. These heavily elastically bent samples almost exploded on fracture, producing numerous very small pieces of flying glass.

3. Abraded samples showing the smallest fracture loads often only broke into two pieces. These could be fitted together exactly. Samples with higher fracture loads were increasingly fragmented as the fracture stress rose; that is, as the stored elastic energy at fracture was higher.
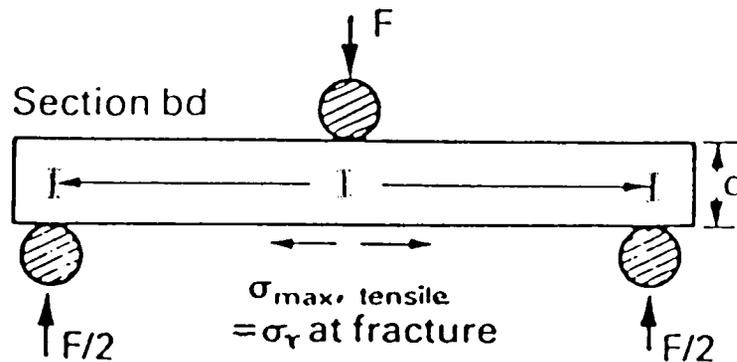

FIGURE 1. Schematic of three-point bending fixture, where F is the applied load, l is the distance between loading points, d is the thickness, and b the width of the sample (slide). The equation for calculating the maximum tensile stress in the surface, i.e., the modulus of rupture is

$$T_r = 3Fl/2bd^2$$
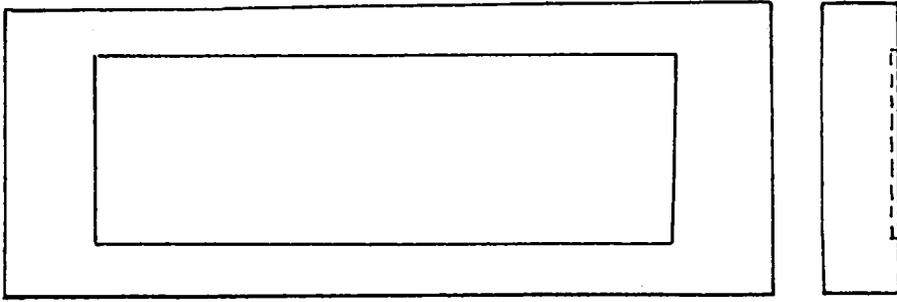
FIGURE 2. A simple holder for scoring a slide surface on 400-grit SiC abrasive paper.

LOW CARBON STEEL: METALLURGICAL STRUCTURE VS. MECHANICAL PROPERTIES

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Institute for Materials Science and Engineering
National Bureau of Standards

PREREQUISITE: A knowledge of phase diagrams and basic metallurgical polishing techniques.

OBJECTIVE: To provide a low-cost, simple experiment for either demonstration purposes or as a laboratory experiment that will teach the student the importance of the thermal-mechanical history of a metallic alloy in determining that material's mechanical behavior.

EQUIPMENT: Several metal hairpins, a candle. a propane torch, matches, a bucket of water, a nonflammable surface, pliers, and (optional) access to a metallurgical laboratory with polishing wheels and cameras.

PROCEDURE: (a) Bend one of the hairpins in the middle to show how ductle it is in the as-received condition.

(b) While holding one of the hairpins with a pair of pliers, heat up a portion of it (the middle is most convenient) with the propane torch as shown in Figure 1a until it is "red hot" and then quench the hairpin quickly by dropping it into the bucket of water.

****
SAFETY WARNING: THE METAL IS VERY HOT EVEN WHEN NOT RED IN COLOR.
THEREFORE, HANDLE IT ONLY WITH THE PLIERS!!
****

Test the ductility of the hairpin by bending it after this high temperature heat treatment. What has happened? If you have a high temperature thermocouple (e.g., Type K: Chromel-Alumel or Type S: Platinum-10% Rhodium) measure the temperature of the torch flame.

(c) Take a second hairpin and heat treat it with the propane torch as performed in (b), followed by a fast quench. Before testing its ductility, place the heat treated region over a burning candle as shown in Figure 1b for a couple of minutes and then quench in water. If you are able, measure the temperature of the candle flame. Test the ductility of the hairpin as before. Has anything changed? Why or why not? Hint: Compare the above two experiments with respect to the iron-carbon phase diagram shown in Figure 2. (The hairpin is a low carbon steel containing approximately 0.10 wt.% carbon, originally in a rolled condition).

(d) If you have access to a metallurgical polishing laboratory, make cuts of the heat treated regions of the hairpins used in steps (b) and (c) and observe their microstructures after mounting, polishing, and etching them. Compare these with the "as-received" microstructure. A good etchant to use is a 1-2 vol.% solution of nitric acid in ethanol.

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SAFETY WARNING: MIXING AN ACID WITH A SOLVENT (LIKE ETHANOL) IS
VERY EXOTHERMIC!!! THEREFORE, ALWAYS ADD THE ACID TO THE SOLVENT!
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*********************************************************************
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Can you see a relationship between the microstructure and the ductility of the hairpin?
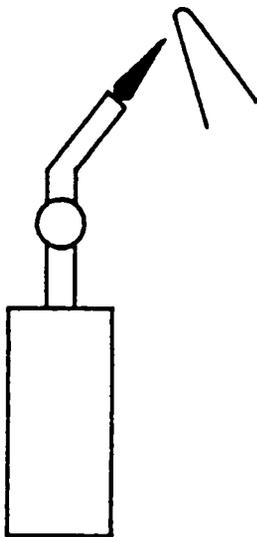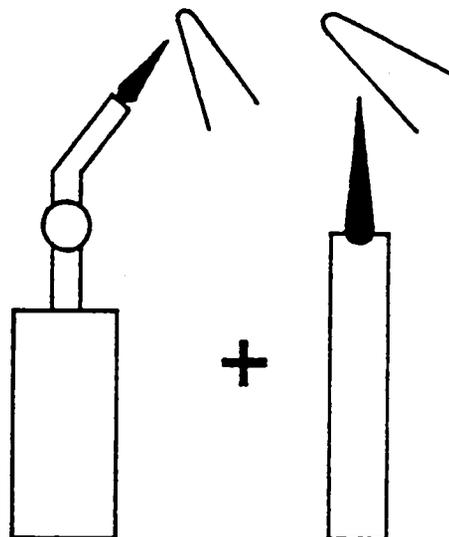
Figure 1a   Figure 1b
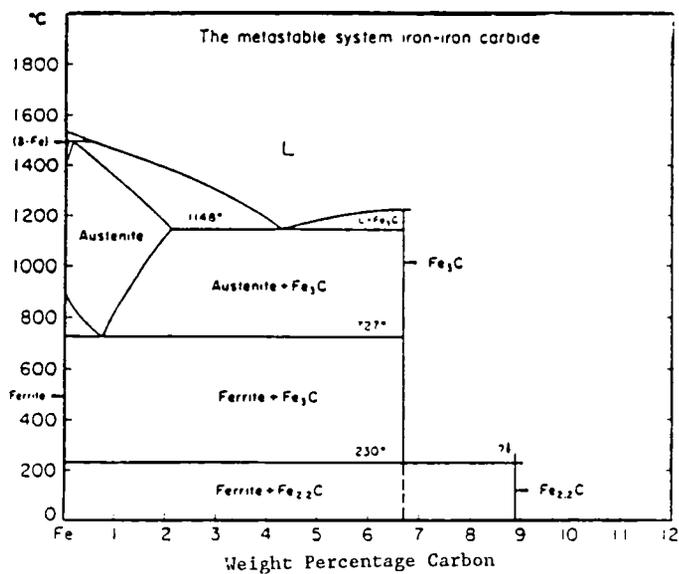
Figure 2

In the "as received" condition. Figure 3 shows that the hairpin was initially prepared from a rolled sheet of low carbon steel that received a substantial reduction in cross sectional area (probably close to 90 % as judged by the small thickness of the highly elongated grains). The absence of non-elongated regions indicates that this material either received no subsequent heat treatment or only a short low temperature stress relief anneal.

While heating the hairpin to a bright red glow in the propane torch (the temperature will reach approximately 1000 degrees Centigrade), the steel will transform to the high temperature face centered cubic (austenite) phase: refer to the metastable phase diagram in Figure 2. On quenching, this steel will change to a body centered cubic structure, but will do so via a martensitic transformation (hence, the sharp needles observed in Figure 4, which show the microstructure for this condition). It's interesting to note that since the habit plane of this martensite is the (111) plane of the original austenite, sometimes one may observe the appearance of hexagonal networks of martensite plates. The internal structure of these martensite plates (needles) is highly dislocated (and therefore highly stressed). The brittleness of the material in this condition is a result of these high residual stresses caused by the phase transformation.

Figure 5 shows the microstructure of the hairpin after aging the martensitic structure in the candle (temperature of approximately 600 degrees Centigrade). During this treatment the substructure of dislocations is slowly eliminated (thereby relieving the stress associated with them) and the material undergoes recovery. The material is now referred to as being in the "tempered" condition. As this tempering process continues, the relatively high carbon content of the martensite is reduced and both metastable cementite, Fe_3C, (dark regions in Figure 5) and ferrite (light colored regions) form, initially at the plate boundaries.

MICROSTRUCTURE OF HAIRPIN "AS RECEIVED"
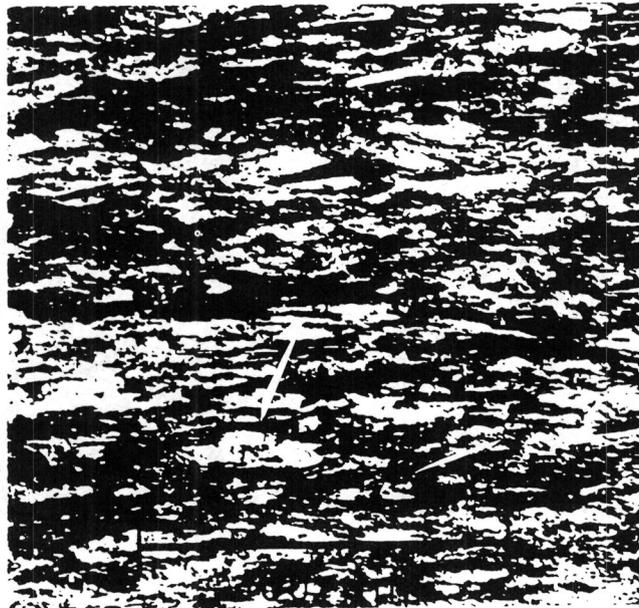
Figure 3

Original figure not available.

MICROSTRUCTURE OF HAIRPIN "HEAT TREATED"
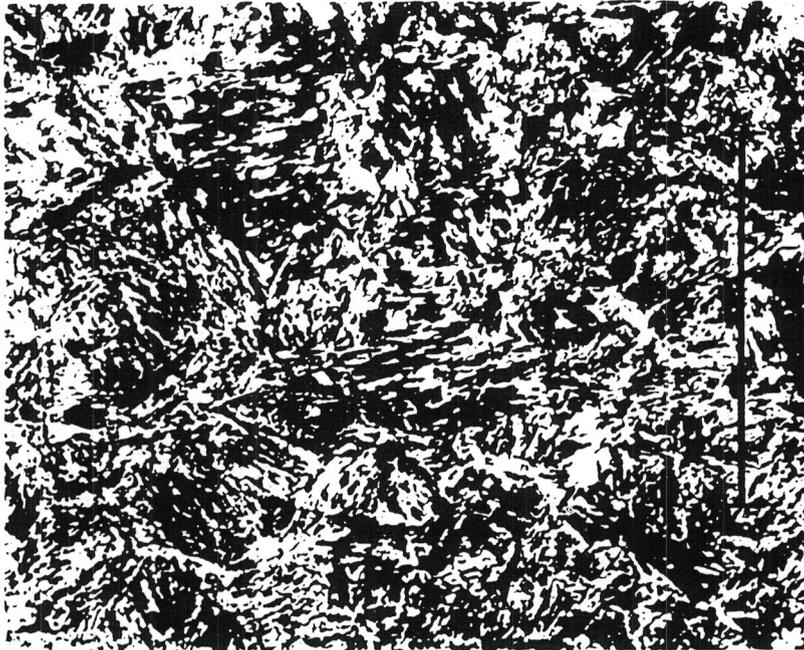

Figure 4

MICROSTRUCTURE OF HAIRPIN "TEMPERED"
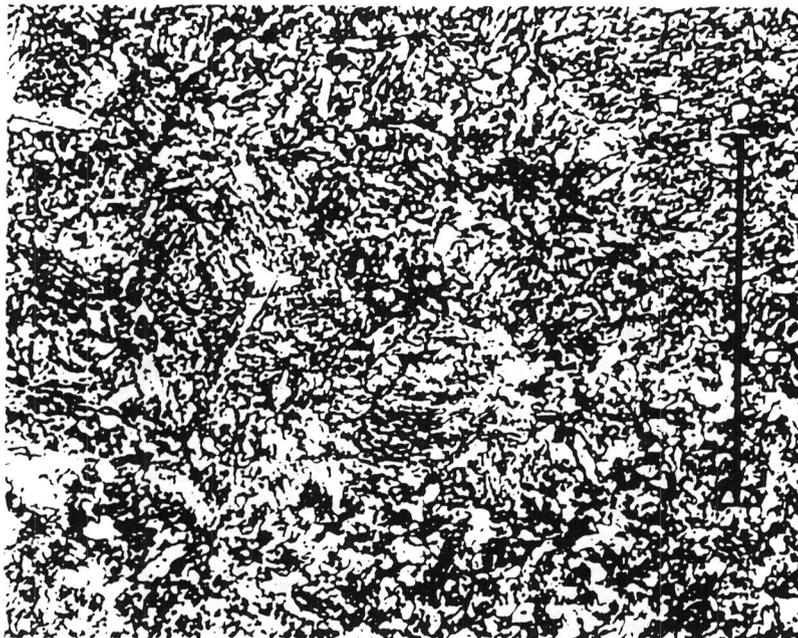
Figure 5
Original figures not available.

THE KELVIN WATER-DROP EXPERIMENT

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PREREQUISITE: This is an experiment originally designed and performed by Lord Kelvin (William Thomson) in the late 1800's to demonstrate the creation of an electric potential simply by means of dividing up a body of flowing water. A basic understanding of electricity is a prerequisite together with a curiosity and desire to find an explanation for the unexpected.

OBJECTIVE: To demonstrate the power of electrical forces in a material as common as water and to help teach the student that even simple, well understood phenomena sometimes present unexpected results that, at first thought, defeat explanation.

EQUIPMENT: Two (or three) large coffee cans, two meters length of copper tubing, two (or three) small tuna fish cans with their tops and bottoms removed, two (or three) glass jars with lids; one bucket, one copper T-joint, one shut-off valve, scrap wood, water, paper towels, and (optional) a voltmeter.

PROCEDURE: (a) Construct a wooden frame to support the apparatus shown in Figure 1: Bucket (P1) should be suspended by strings above pails (P2 and P3) as shown, with tuna fish cans (C2 and C3) connected by means of copper tubing lengths (W2 and W3) to these pails. Good electrical connections should be made at each end of these tubes, and the tubes should be located near each other but not touching. A water cut-off valve under P1 is helpful, and pails P2 and P3 should be electrically isolated from each other by sitting on top of glass jars.

(b) Fill P1 with water, open the cut-off valve to let water flow down the tubes and observe what happens. Measure the voltage between the pails as a function of time. Is there any change? Is the water flow diverted at any time? What about its width? Adjust the height of the water flow above the cans C2 and C3 to change the relative position where the flow breaks up into drops. Is there any relationship to the creation of sparks observed? Hang two copper plates loosely from tubes W2 and W3 so that they are parallel and free to move and form a capacitor with a large surface area. What happens? Why?

(c) If you are indeed adventurous, modify the apparatus to include three pails as shown in Figure 2. How does this modify the effect observed in the first apparatus? If you have three voltmeters, simultaneously monitor the electrical potential between each pair of pails P2, P3, and P4. Plot these voltages versus time on the same graph paper. Are they related?

THE KELVIN EXPERIMENT
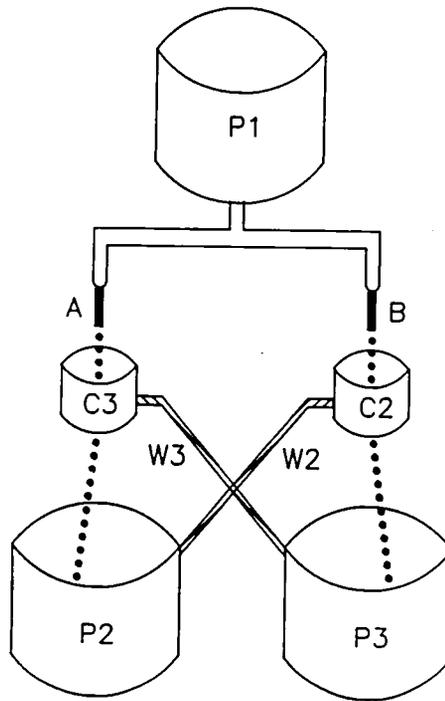
Figure 1

# A THREE-PAIL KELVIN EXPERIMENT
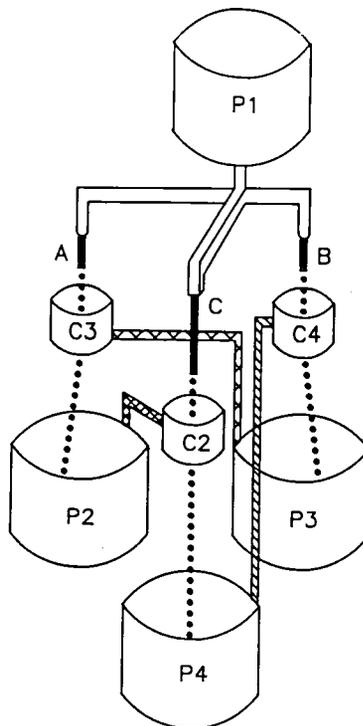
Figure 2

INSTRUCTOR'S NOTES

The experiment in (b) will result in the generation of electrical sparks between W2 and W3, the frequency and strength depending on the distance separating them. However, it is critical that the water flows be adjusted so that the water forms droplets near (or inside) the cans C2 and C3 (don't let the drops hit these cans).

The water initially separates into two flows of equal potential underneath P1. Sometime shortly after starting the flow, one of the water drops formed from flows A or B will possess an extra electron. Let's say it occurs under flow B. This drop will fall into P3 giving it a negative charge. Since C3 is connected to P3, it will develop a negative charge which will exert an electical (repulsive) force on the electrons in the water in flow A. This force will result in the formation of drops under flow A with a deficiency in electrons and therefore will be positively charged. P2 (and therefore C3) will become positively charged and exert an attractive force on the electrons at the end of water flow B as it breaks up into drops, thereby creating more drops on this side that are negatively charged. Consequently, P3 develops a larger negative potential, which causes an increased repulsive force on the electrons at the end of flow A and results in more drops forming under flow A with a positive charge. The potential difference between P2 and P3 continues to build up with time until it exceeds the capacitive breakdown potential between them, and a spark will be created (usually between W2 and W3—the closest points) to allow charge to flow between them. As charge builds up in cans C2 and C3, the attractive force between them and the drops falling through them will result in a greater deflection of the water streams.

By hanging copper plates from W2 and W3, a greater charge will build up between them, and the electrical force between them will cause them to move toward each other. On touching (if close enough) the plates will swing away from each other (by gravity) and one will begin to observe the coupled oscillator phenomena (with the transfer of kinetic energy back and forth between the plates).

The configuration shown in Figure 2 will create a three phase alternating potential, but it is more difficult to get working.

REVEALING THE MICROSTRUCTURE OF MATERIALS
James A. Nelson

PREREQUISITE KNOWLEDGE:
General High School Education; Average Manual Dexterity

OBJECTIVES:
Demonstrate how the microstructure of materials may be revealed by abrasive polishing and chemical etching.

Illustrate how microstructural information is used to monitor manufacturing processes, provide in-depth inspection and perform failure analysis.

EQUIPMENT REQUIRED:

Low Speed Saw	BUEHLER 11-1180-160
Specimen Polisher	BUEHLER 69-1000-160
Drill Fixture	BUEHLER 69-1550
Hand Drill, 1/4"	
Microscope, Metallurgical type	
TV Camera and Monitor (Optional)	

SUPPLIES:

- Test Material:
  Bare plated through-hole printed wiring board samples, double sided, 1" X 3-3/8" (standard IPC Test Coupon Configuration)

- Cutting Fluid

- Cold mounting resins and molds

- Abrasive papers, 2-7/8" dia. assortment

- Polishing cloths, 2-7/8" dia. TEXMET® and MICROCLOTH®

- Abrasive, diamond paste, 6 micron

- Abrasive polishing alumina, 0.05 micron

- Abrasive extender

- Distilled or deionized water

- Ammonium hydroxide

- Hydrogen peroxide, 3%

- Alcohol, methyl (methanol)

SOURCES:

Specimen preparation equipment	Author's company
Supplies for above	Author's company
Microscope, TV camera and monitor	Author's company
Ammonium hydroxide, 3%	Chemical supplier
Alcohol, reagent grade	Chemical supplier
Hydrogen peroxide, 3%	Drug store

PROCEDURE:

Introduction: Microstructural Analysis is the procedure used to reveal the internal microstructural details of a material or part by sectioning and polishing the cut surface so that it may be examined under a suitable microscope. When this surface is correctly prepared, the observer is able to observe, evaluate and measure various microstructural features. The information obtained helps the investigator to assess the soundness of the material or determine if certain processes such as plating have been correctly performed. Microstructural Analysis may be a sole means of investigation but it is more often performed in concert with other test procedures.

Test Material: A printed wiring board has been selected as a test material because it contains both metals and nonmetals that have a distinct microstructure, and because this technique is used throughout the electronic industry as a key quality control tool. The three principal component materials in printed wiring boards are

> Glass/epoxy laminate faced with copper foil
> Copper, deposited by both electroless and electrolytic plating
> Tin/lead solder, eutectic

Printed wiring boards are manufactured by a placing a photographic image of the circuitry including holes and the pads that surround them onto laminate that is covered with copper foil. The circuitry is produced by a series of plating and stripping operations that must be carefully controlled. Because the process has many steps and many parameters to control, there are numerous potential sources of defects. Microstructural Analysis (metallography) provides an in-depth visual analysis of the wiring board cross-section that helps monitor the manufacturing processes and prevents economic loss due to rejectable defects or failure to meet design specifications.

Sample Preparation :

(1.)Sectioning (cutting): The printed wiring board coupon is attached to the arm of the low speed saw by inserting it into a chuck and aligning the coupon with the abrasive blade so that the cut will just miss the test holes that are to be examined. A second cut is made, if required, to produce a sample that is not more that 1/2" high. Refer to the supplemental literature for additional details.

(2.) The cut sample is thoroughly cleaned to remove cutting fluid and moisture that could prevent the mounting resin from adhering to the sample surface.

Mounting: Using plastic clips as a support, the sample is placed into the cavity of a 1-1/4" dia. mold. The mounting resin is measured and mixed according to the manufacturer's instructions and cast into the mold cavity taking care that the resin does not topple the sample or push it to one side of the mold. When cured, the mounted sample is removed from the mold.

Grinding: The cured mount is placed into the alignment fixture and a hole is drilled into the back face (opposite the sample) to allow attachment to the polisher. A 240-grit silicon carbide abrasive disc is next attached to a glass platen and placed into the polishing bowl. A modest amount of tap water is applied to the disc and the sample is placed into the bowl. The tip of the polisher arm is inserted into the hole that was drilled into the back of the sample mount. Settings for speed, time and pressure for the grinder/polisher are detailed in the MINIMET METAL DIGEST ™under Preparation Procedures, sequence A, page 8. (3) The procedure is repeated for each of the successively finer steps (320, 400 and 600 grit). The polished sample must be rinsed between each grinding step and thoroughly washed at the conclusion of this sequence.

Polishing:

A. Using a similar set-up procedure, the specimen is next polished using 6-micron diamond abrasive paste applied to an unnapped (TEXMET®) cloth for about 2 minutes with the addition of a

modest amount of liquid extender.The sample is washed thoroughly and dried at the conclusion of this step.

B. The sample is final polished for about 30 seconds on a napped cloth (MICROCLOTH®) that has been charged with 0.05 micron alumina polishing suspension. At the conclusion of this step, the sample is again washed, rinsed with alcohol and air dried.

Examination:

A. As-polished: The polished specimen is examined under the microscope by scanning the entire area at 50-100 X magnification. This cursory examination will reveal very little detail in the metallic layers because the sample has not been etched. Gross defects, if present, will be seen, but finer defects will be masked by the normal smearing of the softer metals. The woven glass pattern may be faintly visible.

B. Etched Examination: The polished sample is etched with a solution consisting of equal parts of concentrated ammonium hydroxide and hydrogen peroxide (3%). Using a cotton swab, a copious amount of etchant is applied to the polished sample for approximately 3-5 seconds. The sample is then immediately rinsed with tap water to stop the etching action, followed by a rinse in alcohol and warm air dry. Examination will reveal a lightly etched condition with microstructural details not too clearly visible due to residual deformation. To remove remaining residual deformation, repolish the sample on the final polishing cloth for about 30 seconds, followed by a rinse and dry as before. The sample should then be re-etched for a longer duration (about 6-8 seconds), washed and dried as before. Examination will reveal clearer details than were visible after the initial etching, revealing distinct interfaces between the various plated layers and the actual microstructure of the copper and tin/lead layers.

C. Analysis: The polished and etched sample should be examined thoroughly at lower magnifications (50X-100X) first and in selected areas at higher magnifications to more clearly identify possible problem areas. If the microscope has a camera, photomicrographs may be taken to record the results. A typical photomicrograph is shown as an example.

D. Interpretation: The two photomicrographs that are shown were made from the same polished sample but at two different magnifications to illustrate the nature of the information that may be obtained. (See figures.)

- 50X: Illustrates the value of low magnifications to obtain view of the entire through-hole cross-section.
- 200X: Illustrates the use of higher magnifications to obtain closer look at areas that appeared suspicious at the lower magnification

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1. James A. Nelson, *"Basic Steps for Cross Sectioning PCB's"*, INSULATION/CIRCUITS, May, 1977, Reprint No. 26.

2. *"ISOMET® Low Speed Saw Sectioning of Materials,"* BUEHLER METAL DIGEST®, Vol. 22, No. 1, 1983

3. *"MINIMET® Polisher/Grinder,"* BUEHLER METAL DIGEST®, Vol. 20, No. 1, 1981

**Entire through-hole
at 50 x mag.
(As polished)**

Laminate ――――――→

Delamination ――――――→
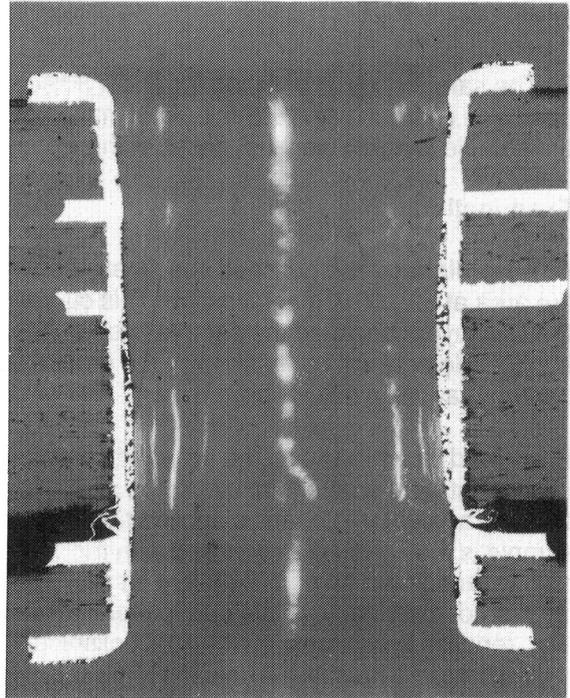
**Knee of the
though-hole
at 200 x mag.**

**Interlayer
with Etchback** ――――――→

Solder ――――――→

Etched

**Ammonium Hydroxide-
Hydrogen Peroxide 3%**
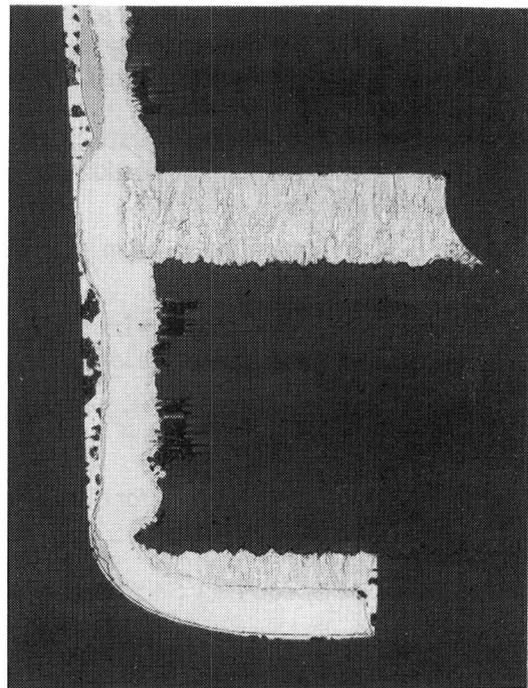
COMPOSITES

Fiberglass Hand Laminating Process

Battelle Northwest - 1987
Richland Schools, Richland Washington

Key Words: Composite, glass fibers, epoxy resin, catalyst, exothermic reaction.

Prerequisite Knowledge:

In the study of polymers it is important to know about thermoset and thermoplastic polymers. For the students to better understand this experiment, they will need to know that epoxy resins when reacted with a catalyst, form a thermoset polymer. The chemical reaction that takes place as the students mix these compounds together causes a special polymer bond known as crosslinking. It is because of this crosslinking that the tough, rigid properties of a thermoset polymer occur and are useful in this experiment.

Learning Objective:

The student will be able to comply with the procedure for making this composite material.
The student will be able to make a fiberglass composite to apply and test the concept of combining two different materials to obtain a new material.
The new material will exhibit new and better properties than the original materials.
The student will understand the reason for combining materials to make a composite.

Materials/Equipment:

1. Fiberglass
2. epoxy resin and catalyst (hardener)
3. 4-6 oz. paper measuring cup
4. brush (1 inch)
5. polyethylene film 8-10 in. sq. (clear plastic bag)
6. acetone
7. tongue depressor for a stir stick
8. plastic gloves

Procedure:

1. Cut five to six 4 X 4 in. fiberglass squares with scissors.

2. Measure two ounces of epoxy resin into paper cup and proceed to follow directions provided by the manufacturer. (See Instructor Notes.) Thoroughly mix contents with tongue depressor for 3 -5 minutes.

3. Place polyethylene film on tabletop.

4. Pour a small amount of resin onto the polyethylene surface. (Note: resin does not stick to polyethylene.) Spread to 4 X 4 in. area with brush.

5. Place one fiberglass 4 X 4 in. mat onto resin.

6. Dip brush into resin and begin to brush the resin on the fiberglass mat from the middle outward. The fiberglass will absorb the previously poured resin as you brush. Apply only enough resin with the brush to saturate the fiberglass.

7. Place second fiberglass mat (dull side up) onto the first layer. Apply resin with brush, working from the center out to prevent air bubbles. Add only enough resin (with the brush) to saturate fiberglass.

8. Repeat process with each additional fiberglass laminate until desired thickness is achieved.

9. Cover the fiberglass laminate with a polyethylene sheet. Apply a heavy, flat object to the top of the composite to compress the fibers and drive off excess resin.

10. Observe the contents of the cup and brush to verify that the resin is curing. The cup will feel warm from an exothermic reaction that is taking place. An increasing odor will be noticed as the reaction evolves.

11. Clean the epoxy brush with acetone. Discard the cup and clean the tabletop if necessary.

12. Allow the resin in the composite to cure. Then the composite will be ready to remove easily from the polyethylene sheets to be examined and/or tested. Overnight should be ample time for curing the material.

INSTRUCTOR NOTES:

It is significant to know that on the market there are many different epoxy resins and hardeners available. When mixing these compounds, it is very important to follow the ratios suggested by the manufacturer of the resin. A data sheet to instruct the user normally accompanies the resin. Carefully follow these instructions to obtain a desired reaction and thermoset polymer.

CAUTION

Two ounces of epoxy resin is recommended in this experiment to keep the quantity of materials at a minimum but also to limit the reaction if the compounds are mixed incorrectly or a mistake is made. The reaction has been known to get very hot. If this occurs, put on leather gloves and move it to a heat resistant surface and to an area where the air is vented to the outside.

This simple experiment can be enhanced in many ways. Other materials can be used in place of the fiberglass to form the composite (i.e., Kevlar or graphite fibers). Fillers can also be used to make lighter and/or thicker materials. Types of epoxy resins and hardeners can be varied or even completely changed to use other types of polymers as bonding agents in the composites. These suggested enhancements can also be considered as completely different experiments because of the changes that are made. To ensure success, the instructor would need to understand the variations and complications that are involved when working with the vast arrays of polymeric materials.

Testing materials is also another area that can be studied and can be considered an entirely different field of study. Tests can be designed for strength, impact, chemical, and environmental resistance, and reactions to temperature-- to name a few.

Sources of Supplies:

All of the above supplies can be purchased locally. Sometimes the materials can be found in complete kits at your local hardware store. In some cases, it may take some phone calls to locate all the items needed. A good place to check would also be local auto body shops and auto supply stores.

GLASSES AND CERAMICS

Making and Testing Superconductors

Battelle Northwest - 1987
Richland Schools, Richland Washington

Key Words: Superconductor, superconductivity, resistance, degrees Kelvin (°K), ceramic, metal oxides, sinter, ohm, Meissner effect, mole, molar ratio, molecular formula, critical temperature (T_c).

Prerequisite Knowledge:

Superconductors are conductive materials which have an extremely low resistance to the flow of an electric current; that is, they have a theoretical resistance (R) equal to zero ohms. Most materials which exhibit superconductivity possess this property only at very low temperatures. Until 1986, these temperatures were close to absolute zero (0°K) (see instructor note 2). In a recent breakthrough, superconductivity was observed at temperatures slightly above that of liquid nitrogen (77°K). Now the race is on in the scientific community to create a superconductor which is able to conduct electricity without resistance at even higher temperatures. Although the new superconductors are in the developmental stage, someday they may be used in superfast computers, magnetically levitated trains, high-powered electric cars and energy transmission lines which transmit electricity with virtually no power loss.

Follow this procedure to make a ceramic superconductor from three metal oxides: yttrium oxide (Y_2O_3), barium peroxide (BaO_2), and copper (II) oxide (CuO). $BaCO_3$ can be used in place of BaO_2. The molar ratio of the three metals in the complex is 1:2:3, yttrium to barium to copper. The following is one way of expressing the molecular formula for this particular superconductor:

$$YBa_2Cu_3O_{7-x} \qquad\qquad (ideally \quad YBa_2Cu_3O_7)$$

Learning Objective:

1. The student will make a superconductor by employing the following techniques: chemical batching, mixing and grinding (pulverizing), heating, pressing, and tempering.

2. The student will be able to verify the superconductivity of a material through testing by using two methods outlined in the procedure.

3. The student will be able to reason how applicable methods superconductors can be used in our society.

Materials/Equipment:

1. balance
2. furnace capable of achieving 950°C
3. furnace controller to ramp temperature at controlled rates
4. hydraulic press
5. grinding chamber (see instructor note #5), disc mill, rock tumbler, or automated mortar and pestle (standard, hand-operated mortar and pestle may be used).

6. crucible - Alumina or ceramic crucible
7. yttrium oxide, Y_2O_3
8. barium peroxide, BaO_2, (or barium carbonate)
9. copper (II) oxide, CuO
10. die to form superconductor
11. solvent: alcohol, trifluorotrichloro ethane or toluene (a solvent that evaporates quickly is preferable)
12. disposable protective gloves (such as PVC gloves)
13. safety glasses
14. zinc stearate
15. alcohol (EtOH)
16. liquid nitrogen
17. Samarium cobalt magnet (see instructor note #6)
18. micro-ohmmeter for testing purposes (four-point type)
19. tweezers
20. annealing oven capable of achieving 475°C
21. oxygen gas (optional)
22. fume hood
23. particulate mask (recommended)

Procedure: Making Superconductors

1. Make calculations for the batch using the 1:2:3 ratio. The following is an example for a .1 mole batch, which is enough to make several superconductors. To achieve a ratio of 1:2:3 (Y, Ba, Cu), use a molar ratio of 1:4:6 for (Y_2, Ba, Cu).

Ratio	Compound	Molecular Weight (g/m)	Multiplier (for .1 mole)	Mass (g)
1	Y_2O_3	225.81	.1	22.58
4	BaO_2	169.33	.4	67.73
6	CuO	79.54	.6	47.72

CAUTION

Some of the chemicals used in the making of superconductors are toxic. Please check the content of these materials in a chemical safety book. When chemicals are exposed, such as in weighing or open container grinding, the work should be done in a ventilating (fume) hood. Disposable plastic gloves should be worn, and it is advisable to wear a good quality particulate mask for added safety.

2. Weigh the chemicals and place them in a container to go into a grinding chamber or disc mill. If a grinding chamber or disc mill is not available, use a) a rock tumbler with a very hard object such as a chunk of stainless steel or a piece of quartz added to help the grinding process; or b) a mortar and pestle.

3. Add 30 grams of alcohol, trifluorotrichloro ethane (freon) or toluene to aid in mixing.

4. Grind for one hour. The smaller the resulting particles, the better. Grinding the material to the smallest possible size is <u>very</u> important in producing superconductor material.

5. Let the solvent evaporate in a hood.

6. Place powdered mixture in furnace at 900-925°C for 18 hours.

7. Remove from furnace, cool, and repeat steps 3 through 7.

8. After removing from oven the second time, examine the mixture to see if any greenish-colored material is evident (see instructor note #3). If so repeat steps 3 through 7. More than 25% greenish material indicates an incorrect mixture of chemicals, poor chemical quality, or improper oxidation; a new chemical batch is necessary.

9. Grind again to a fine powder--once again, the smaller the particles, the better.

10. Weigh out quantities of the material to make pellets. The quantities are determined by the size of the mold to be used. (We used 12g and 15g quantities to form pellets about $9 \times 17 \times 42$ mm.)

11. Clean the die with alcohol. Lubricate with a very light dusting of zinc stearate.

12. Place the material in the die and distribute it evenly.

13. Put the top on the die and prepare it for pressing on the hydraulic press.

   a. Gradually increase the pressure to 5000 lbs. (area ≈ 1.0 in^2 for a pressure of 5000-6000 lb/in^2.)

   b. Hold the pressure for a couple of minutes.

   c. Let the press gradually release the pressure itself. (The press seems to release in "jerks" when done.)

14. Remove the top part of the die and extract the pellet.

15. Place the pellet in the furnace for sintering.

   a. Heat the furnace at 300°C/hour until it reaches 950°C.

   b. Allow the pellet to sinter at least 8 hours at 950°C.

   c. Cool at 50°C/hour to bring the furnace back to room temperature.

16. Place in an annealing oven at 450°C for 18 hours. If oxygen gas is available, bubble O_2 over the pellets while they are being annealed. After the allotted time, let the oven cool to room temperature with the pellets inside.

17. Remove the pellets from the anealing oven. They are now ready to be tested for superconductivity.

18. Attach the micro-ohmmeter (four-point type) to the pellet and take a resistance reading at room temperature. Now place the pellet in liquid nitrogen so it is submerged and take a second reading. In its cooled state the resistance measurement should read zero ohms. (Readings from this measurement can be erratic due to poor contact or high-contact resistance.

19. Test for the Meissner effect (see instructor note #4) using a Samarium Cobalt magnet and liquid nitrogen.

    a. Place the superconductor in a pool of liquid nitrogen (a Styrofoam cup cut down to an inch high works well in containing the liquid N_2 when working with the superconductor; the pellet can be placed on a small brass block acting as a pedestal while in the liquid N_2).

    b. Allow the superconductor to cool for a few minutes. Continue replenishing the liquid nitrogen supply around the pellet as the N_2 dissipates.

    c. With the tweezers, carefully place the magnet so that it is just above the superconductor. The height at which the magnet will remain suspended in air varies depending on the strength and size of the magnet--the smaller and stronger the magnet, the better. Once the magnet is balanced above the pellet, it can be set into a spinning motion with the flick of a finger or tweezers.

Instructor Notes:

1. This experiment is lengthy. It would be best to divide a classroom into groups of two, three or four students each, or to let a special interest group do the experiment initially. It may take several tries before a superconductive material is made, so allow plenty of time for experimentation. A highly motivated group of students could go through the process first and identify the critical areas of the process to the class before the class attempts the experiment. The grinding process (see instructor note #5) is of crucial importance. Even though the students may be eager to make this material, emphasize that they must perform each step carefully. This will better ensure that they successfully produce a superconducting material the first time through the procedure.

2. Superconductivity, in which the resistance (R) is virtually equal to zero, was first discovered in 1911 by Heike Kamerlingh Onnes while observing mercury at liquid helium temperatures (4°K, -452°F). The critical temperature (T_c) at which a material is superconductive has remained very low since that first discovery, rising only about 4°K per decade with research. By 1973, the best of the superconductors possessed a T_c of 23°K (-418°F). The discovery in 1986 of a superconductor with a T_c greater than the temperature of liquid nitrogen (77°K, -321°F) was a giant step toward bringing the whole process closer to the realm of practicality. Cooling with liquid helium is expensive, whereas the cost of a gallon of liquid nitrogen is comparable to the cost of a gallon of milk.

3. The greenish material mentioned in step 8 of this experiment has been seen in several studies done on this particular superconductor. The greenish material is a non-superconductive phase which has the composition of Y_2BaCuO_5. The black phase is the desirable super-conducting material, approaching the ideal composition of $YBa_2Cu_3O_7$.

4. The Meissner effect is a phenomenon which all true superconductors exhibit while in their superconductive state. When cooled to its proper temperature (the critical temperature, T_c), the superconductor repels all magnetism regardless of polarity. The classic picture of a magnet suspended in midair above a superconductor is a result of the Meissner effect.

5. The mixing and grinding of these chemicals is very important. The smaller the particle size, the better the chance superconductivity will occur. Any number of grinding methods can be used, preferably automated. If hand grinding is necessary, be certain to grind as long as possible. One way to do this would be for a group of students to share the work, each taking a ten to twenty minute turn at grinding. If a rock tumbler is used, operate overnight and check the consistency of the material the next day to see if it is finally powdered. The other automated systems should be operated for a minimum of one hour.

6. Samarium cobalt magnets have an extremely strong magnetic field for their size. This is important because common magnets are generally bulky and have weak magnetic fields. The weight of the magnet can overcome the force of the electric field and the levitating [Meissner] effect will not be seen. Samarium Cobalt magnets can be purchased commercially; these same magnets are commonly used in light-weight head phones, in case you have an old pair to disassemble.

Resources:

Heppenheimer, T.A. "Superconducting: The New Billion Dollar Business." High Technology, July 1987: pp. 12-18.

Maranto, Gina. "Superconductivity: Hype vs. Reality." Discover, August 1987: pp. 22-32.

"Ceramic Superconductors." Advanced Ceramics Materials Special Supplementary Issue, July 1987.

For those with low budgets, contact a local college chemistry department or industrial laboratory for the materials and other help. The materials can be ordered from the following vendors:

General Vendors:

Allied Fisher Scientific, 50 Fadem Road, Springfield, NJ, 07081. Phone # (201) 379-1400.

Mortar and Restal ($75-$200), Alumina crucibles ($20-$30 ea.)

VWR Scientific, P.O. Box 7900, San Francisco, CA 94120. Phone # (415) 468-7150.

American Scientific Products, 1430 Waukegan Rd., McGraw Park, IL 60085. Phone # 1 (800) 325-4520.

Chemical Vendors:

Alfa Products, Morton Thiokol, Inc.; 152 Andover St., Danvers, MA 01928. Phone # (617) 777-1970 and 1 (800) 343-0660.

Yttrium oxide (99.99%), 250g, at $80; Zinc stearate, 1 Kg at $15; Barium peroxide, 500 g at $18; Copper II oxide, 1 Kg at $56; Ethanol, anhydrous, 500 ml at $13.

Aesar, Johnson Matthey, Inc., P.O. Box 1087, Seabrook, NH 03874. Phone # (603) 474-5511 and 1 (800) 343-1990.

Aldrich Chemical Co., Inc., 940 W. Saint Paul Ave., Milwaukee, WI 53233. Phone # 1 (800) 231-8327.

It is assumed that the large pieces of equipment are available in your school or from a source in your community; otherwise they will need to be purchased. Some equipment can be improvised. Dies can be made by the students or machined in the school machine shop.

If the school is without a press, an appropriate small die can be made to compress the pellets by striking the die with a five pound sledge hammer. It would be best if the die were tempered after machining, but even untempered steel will be able to make a number of pellets before it mushrooms out and becomes unusable. The reason for pressing the pellets is to compact the chemicals as close together as possible and to have a solid piece to place into the furnace. This improvision can create pellets similar to that of a hydraulic press.

Many institutions (i.e., hospitals, laboratories, doctors, colleges, industries) use liquid nitrogen. Contact them for their source or to request a donation from them.

See instructor note #6 for rare earth oxide magnets.

RESOURCES:

(Description of a service or product is not intended as an endorsement. An * indicates the entry is an expansion of an earlier listing.)

*SCI announces production of two high-T_c powders. (1) $Y - Ba - Cu - O$ CP (tm) superconductive powder produced by co-precipitation process. All powder is 95% or better primary orthorhombic phase, average particle size 1 micron. T_c approximately 90 K or better with 6 K transition width, without oxygen anneal. (2) $Y - Ba - Cu - O$ SS (tm) superconductive powder produced by "solid state" process, T_c approximately 82 - 83 K. Other powder compositions, pressed pills and other shapes available upon request. Contact Superconductive Components, Inc., 1145 Chesapeake Ave., Columbus, OH 43212; telephone (614) 486-0261.

Edmund Scientific is marketing superconducting ceramic discs for educational laboratory demonstrations. Manufactured by Tektronix Co., the disc is composed of $Y - Ba - Cu - O$; T_c is 83 K. Kit, including disk, holder, instructions, and bibliography, is $20; two or more kits, $18 each. Contact Edmund Scientific Co., 101 E. Gloucester Pike, Barrington, NJ 08007; telephone (609) 573-6250 or 547-3488.

Proceedings of the Beijing International Workshop on High Temperature Superconductivity and Proceedings of the Adriatico Research Conference on High-T_c Superconductors, ICTP, Trieste, Italy, 6 - 8 July 1987, are now available from World Scientific Publishing Corp. World Scientific is also publishing the Proceedings of the Drexel International Conference on High-T_c . Contact: World Publishing Co., P.O. Box 128, Farrer Road, Singapore 9128; telex RS28561 WSPC; telefax 2737298; telephone 2786188; or, in the U.S., World Scientific Publishing Co., 687 Hartwell Street, Teaneck, NJ 07666; telephone (800) 227-7562.

Los Alamos Nat'l Lab has been named as part of the national superconductivity initiative. Its special charter is to explore and develop ways for DOE labs to form constructive partnerships with industry. Contact Fred Morse (505) 667-1600.

Falmouth Associates has begun a comprehensive study of the starting materials used in the synthesis and fabrication of high-T_c superconductors. The study surveys the current worldwide reserve and production capacity of relevant raw materials, details how they are refined and prepared, describes and assesses lubrication routes, and forecasts demand. Subscribers will obtain a complete business analysis of the materials as well as information on suppliers, fabricators, new business opportunities, etc. For more information, contact Dr.

Hugh D. Olmstead, V.-P, Falmouth Associates Inc., 170 U.S. Route One, Falmouth, Maine 04105; telephone (207) 781-3632; telefax (207) 781-4383; telex 294139.

*Chemistry of High-T_c Superconductors is a new book published by the American Chemical Society. This multidisciplinary collection of articles presents the most current information in developing technologies for commercial uses of high-T_c superconductivity. Editors are David L. Nelson, M. Stanley Whittingham, and Thomas F. George. Cost in U.S. and Canada, $64.95; overseas, $77.95. For more information on ACS Symposium Series No. 351, contact ACS, Distribution Office Dept. #246, P.O. Box 57136, West End Station, Washington, DC 20037.

Superconductivity: The Complete Resource Guide is published by The Bureau of National Affairs, Inc., BNA describes it as "the first and only comprehensive, current sourcebook of names, addresses, projects, issues, and specialized guidance focusing on the superconductivity industry." The Complete Resource Guide is made up of several services: a reference book, including lists, directories, and calendars, overviews, texts, and how-to guides, and a hotline for updates. Charter subscription rate is $445; after October 30, $595. For more information, write The Bureau of National Affairs, Inc., Circulation Department, P.O. Box 40947, Washington, DC 20077-4928; or call the BNA Response Center at (800) 372-1033.

Demo kits to help students discover superconductivity have been developed by KnutSoft Knowledge Systems. Intended for ninth-grade level and above, each kit contains a superconducting wafer, a small magnet, and 20 booklets explaining the history of superconductivity, superconductor applications, recent breakthroughs, and current problems. A bibliography is included. For further information, contact KnutSoft Knowledge Systems, 219 Los Cerritos Dr., Vallejo, CA 94589; telephone (707) 648-0779. $49

Lake Shore Cryotronics has just won an I-R 100 award for its DT-470 Series Temperature Sensor. "Each DT-470 series sensor incorporates a proprietary silicon diode chip with temperature characteristics that are so stable and uniform from chip to chip that they are the first mass-produced interchangeable sensors available for use in the critical cryogenic temperature range from 1.4 K to 475 K. ...Used in conjunction with control instrumentation, the sensors are ideal for a variety of field applications where temperatures from 1.4 K must be achieved and precisely maintained." For more information, contact Jeff Bergen, VP, Marketing; Lake Shore Cryotronics, Inc.; 64 East Walnut St.; Westerville, OH 43081; telephone (614) 891-2243.

MARKET GROWING FOR HIGH SCHOOL KITS

There are now at least four companies marketing superconducting levitation demonstration kits in the United States. The least expensive costs $25 and will be available from American Chemet. A kit costing $49 is available from Knutsoft Knowledge Systems. A third kit is available for $65 from a Denver firm. A fourth company, also based in Denver, intends to offer several kits by the end of this year.

Because demand is so high for his "1-2-3 Levitation" kits (which cost $25), Arthur Ellis of the University of Wisconsin said he will soon be handing the production over to American Chemet, based in Deerfield, Illinois. About 1000 high schools have been reached with the kit, and some are going overseas. Included in the kit is a superconducting ceramic disc (and a recipe for it), magnets, background history of super-conductivity, an instruction booklet that includes an overhead projector demonstration, a list of periodicals for research, and safety warnings. (Arthur Ellis, Chemistry Department, University of Wisconsin, Madison, WI 53706.)

Knutsoft Knowledge Systems of Vallejo, Calif., is marketing a $49 kit that includes a disc and magnets, an introduction to the topic, a periodical list, a lab guide and a demonstration instruction booklet. (Knutsoft Knowledge Systems, 219 Los Cerritos Dr., Vallejo, CA 94589.)

Laboratory Specialists Inc., of Denver, Colo., markets two kits: a $100 deluxe kit which contains a disc, two magnets, experimental samples, a resistivity measurement device, and a manual including experiments, history and safety warnings. Its $65 basic kit contains two magnets, a disc and the manual. (Laboratory Specialists, Inc., 2235 Hudson St., Denver, CO 80207.)

SuperconTech (formerly Oxitec Inc.), also located in Denver, has developed three kits, including an introduction kit for the junior high level costing $65; a senior high level kit costing $120; and a high school/introductory college kit costing $250. The company is developing a fourth kit, priced at $1000, which will contain a SQUID and Josephson junctions. The kits will be on the market by mid-December. (SuperconTech, 821 Acoma St., Suite 200, Denver, CO 80204.)

SUPERCONDUCTIVITY KITS AVAILABLE

A "levitation kit" is now being offered for sale to high school science teachers for the purpose of demonstrating the Meissner Effect. The Institute of Chemical Education (ICE) a nonprofit scientific organization headquartered in Madison, Wisconsin is making the kit available at their cost, $25.00. The kit concept was first introduced to high school science teachers at an ICE workshop held at the University of Wisconsin during the summer. The reception to this new educational aid has been good. According to Art Ellis, University of Wisconsin Chemistry Department, "Ten percent of the flyers sent by ICE to more than three hundred high school educators nationwide have already been returned with a check." The demonstration kit includes a one inch diameter disc, one quarter of an inch thick, coated with wax. The wax coating reduces the disc's exposure to water vapor thereby increasing its stability and extending its life. Also included in the kit are two small samarium cobalt magnets, one iron neodymium boron magnet, and three article reprints. The magnets are being provided free of charge by Crucible Magnetics of Elizabethtown, Kentucky. Instructions for performing the experiment using an overhead projector as well as important background information for teachers are contained within the three article reprints. American Chemet Corporation's East Helena, Montana plant will be providing the superconductor to ICE under contract. Until American Chemet completes the scaleup step, the University of Wisconsin labs will be producing the supercon-

ducting discs. Interested high school teachers should contact Art Ellis, University of Wisconsin (Phone: 608-262-0421) or send a $25.00 check or Money Order (payable to ICE) to Institute for Chemical Education, Project 1-2-3, Department of Chemistry, University of Wisconsin, Madison, Wisconsin 53706.

For anyone who is not a high school science teacher and would like to obtain materials to perform the Meissner Effect, Edmund Scientific Company in Barrington. New Jersey is offering a superconducting ceramic disc for laboratory demonstration. Manufactured by the Tektronix Company, the superconducting disc kit contains a one inch diameter, one eighth inch thick 123 disc, complete with foam holder, instructions, and bibliography. By magnetic levitation of a small samarium cobalt magnet at liquid nitrogen temperature (-77 K) the disc will demonstrate the Meissner Effect. The Edmund catalog advises that most local hospital and lab supply firms have liquid nitrogen available.

The price of the superconducting disc is $20. If two or more are ordered the price is $18 each. A samarium cobalt magnet, 5/16" diameter x 1/16" thick, may be purchased separately for $35.00. Order the kits and magnet from Edmund Scientific Company, 101 East Gloucester Pike, Barrington, New Jersey 08007. The catalog numbers for the disc and magnet are A37,446 and A37,447, respectively.

Demonstration of the Meissner magnetic levitation and a low friction magnetic bearing.

AUTHOR

Colorado Superconductor, Inc.
555 Spindrift Ct., P.O. Box 8223, Fort Collins, CO

PREREQUISITE KNOWLEDGE REQUIRED FOR THE EXPERIMENT

None. All experimental procedures, safety and handling precautions, and background information is contained in the instruction manual which is provided with each set of materials in the kit. The table of contents of the manual is reproduced below.

Note that there is especial emphasis placed on safety. While liquid nitrogen is not and cannot be provided with the kit, both sources and safe handling procedures have been detailed. A prior knowledge of handling liquid nitrogen is encouraged.

OBJECTIVES

Demonstration of the Meissner effect through magnetic levitation.

Demonstration of one application of the Meissner effect: The low friction magnetic rotation bearing.

Demonstration of magnetic flux penetration and the Type II nature of ceramic superconductors via the stacking of the superconductor disks.

EQUIPMENT AND SUPPLIES

The Complete Demonstration Kit, which consists of a 1 inch diameter YBCO super-conductor disk, neodymium or Samarium cobalt magnets, nonmagnetic tweezers, and an extensive instruction manual.

Liquid nitrogen. About a pint should be sufficient for one experiment.

A small Dewar flask to hold and transport the liquid nitrogen.

THE MEISSNER EFFECT

One of the properties of superconductors most easy to demonstrate, and also the most dazzling, is the Meissner effect. Superconductors are strongly diamagnetic. That is to say that they will repel a magnet.

A superconductor is fundamentally different from our imaginary 'perfect' conductor. Contrary to popular belief, Faraday's law of induction does not explain magnetic repulsion in a superconductor. When it is in a superconducting state (that is, at a temperature below its critical temperature T_c), a superconductor will not allow any magnetic field to enter it. This is because microscopic magnetic dipoles are induced in the superconductor that oppose the applied field. This induced field then repels the source of the applied field and will consequently repel the magnet associated with that field. This implies that if a magnetic was placed on top of the supercon-ductor when the superconductor was above its critical temperature, and then it was cooled down to below T_c, the superconductor would then exclude the magnetic field of the magnet. This can be seen quite clearly since magnet itself is repelled, and thus is levitated above the superconductor (Figure 1).

This magnetic repulsion phenomena is called the Meissner effect and is named after the person who first discovered it in 1933. It remains today as the most unique demonstration of the phenomena of superconductivity.

PROCEDURE

The shallow dish-like depression in the kit, or a third of an inch high portion of the bottom of a Styrofoam coffee cup can be used for holding liquid nitrogen for the experiment.

1. ACTION: Carefully pour a small amount of liquid nitrogen into the dish or Styrofoam cup until the liquid is about a quarter of an inch deep.

    RESULT: The liquid boils furiously for a short while. Wait until it stops.

2. ACTION: Using the provided tweezers, carefully place the black superconductor
             disk flat in the liquid until its top is just flush with the surface of
             the liquid nitrogen.

    RESULT: Again the nitrogen boils around the disk. Wait until this stops too.

3. ACTION: Again using the tweezers, pick up the provided magnet, and attempt to
             balance it on top of the superconductor disk.

    RESULT: Instead of settling down onto the surface of the superconductor, the
             magnet will simply 'float' a few millimeters above the superconductor.
             This is a demonstration of the Meissner effect.

The new YBCO ceramic materials are Type II superconductors; thus, the Meissner effect
appears to be a bulk phenomena. This can be demonstrated by stacking two or more
superconductor disks. With the addition of each disk, the magnet will be levitated
higher.

If one carefully sets the magnet rotating, one will observe that the magnet continues
to rotate for a long time. This is a crude demonstration of a frictionless magnetic
bearing using the Meissner effect. The rotation speed of a cube-shaped magnet can
be improved by using a plastic drinking straw to blow a stream of air at one of the
edges or corners of the cube. Another way to increase the rotational speed is to cut
out a small rectangular hole in a piece of paper and place the hole over the levi-
tated magnet such that half of it projects above the plane of the paper; and then if
one blows at the part of the magnet above the paper, it can be made to rotate quite
rapidly.

The cubical magnet naturally is slowed by the resistance of air. Consequently, it
can be expected to stop soon. A cylindrical magnet will rotate for much longer since
it is more rotationally streamlined. However, the cubical magnet makes this
demonstration much more graphic. A research group at Cornell University has
demonstrated a frictionless superconducting bearing that rotates at the rate of 1
million rotations per minute. A bearing such as this operating on the Meissner
effect is much more convenient and safe than a conventional magnetic bearing because
of The 'self-centering' nature of the Meissner effect.

SAMPLE DATA
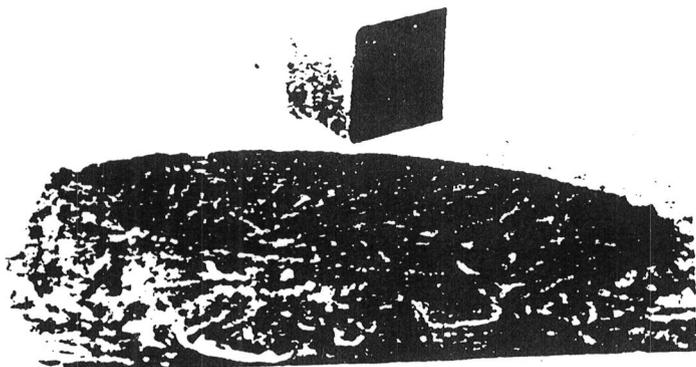
Figure 1   A neodymium magnet levitated over a YBCO disk in nitrogen.
Original figure not available.

INSTRUCTOR'S NOTES

Precautions

1. When pouring liquid nitrogen, be careful to prevent any splashing. Read the section in the manual on safety and handling (pages 10, 11, 28, & 29) before beginning the experiment.

2. Conduct the experiments in a well-ventilated room.

3. Do not touch any items immersed in the liquid nitrogen with your hand until they have warmed to room temperature.

Some Questions

1. Why does the liquid nitrogen boil when you pour it into the dish?

2. Why does it boil when you put the superconductor disk into it?

3. When the nitrogen has evaporated, the magnet stays levitated for many more minutes. Why is this so? Can you think of any other experiments using this fact?

4. If you push the levitated magnet with the tweezers so as to move it across the superconductor, it will resist movement. Why does this happen?

5. How can you improve the operation of the model frictionless bearing in your kit?

This was a demonstration of the Meissner effect. There are many potential applications of this effect, for example: magnetically levitated trains, frictionless bearings, low vibration mounts, etc. Can you think of other applications?

The Critical Temperature of a superconductor (T_c) can be measured by using the Meissner effect. For this purpose the Advanced Demonstration Kit also contains a special device with embedded and characterized thermocouple thermometer.

TITLE

High Temperature Superconductors: Demonstration of zero resistance and the measurement of Critical Temperature T_c.

AUTHOR

Colorado Superconductor, Inc.
555 Spindrift Ct., P.O. Box 8223, Forth Collins, CO 80526

PREREQUISITE KNOWLEDGE REQUIRED FOR THE EXPERIMENT

Previous experience with electrical power supplies and voltmeters is desirable but not essential. All experimental procedures, safety and handling precautions, and background information is contained in the instruction manual which is provided with each set of materials in the kit.

OBJECTIVES

Demonstration of zero electrical resistance in the new ceramic superconductors below their Critical Temperature T_c.

Accurate measurement of the Critical Temperature using the four point probe and the attached thermometer.

For more sophisticated students, measurement of the Critical Current, Critical Magnetic Field, and the reverse ac Josephson effect.

EQUIPMENT AND SUPPLIES

The University Demonstration Kit, which consists of an electrical four point probe device with attached thermometer, a 1 inch diameter YBCO superconductor disk, neodymium and samarium cobalt magnets, nonmagnetic tweezers, and an extensive instruction manual.

Liquid nitrogen. About a pint should be sufficient for one experiment.

A small Dewar flask to hold and transport the liquid nitrogen.

A constant current source, or a power supply operating in the current limited mode. The output should be 0.5 Amp.

An ammeter or second voltmeter (with a current shunt) to measure current flow.

A digital voltmeter with a 10 microvolt resolution to measure the voltage drop across the voltage probes.

For the measurement of the Critical Magnetic Field, a 1 Tesla magnetic field source.

For the reverse ac Josephson effect, an ac signal source with a tunable frequency of up to about 20 MHz and a peak-to-peak signal of 10 Volts.

THE FOUR POINT ELECTRICAL PROBE

When a simple measurement of the resistance of an electrical test sample is performed by attaching two wires to it, one inadvertently also measures the resistance of the contact point of the wires to the sample. Typically the resistance of the point of contact (called contact resistance) is far smaller than the resistance of the sample, and can thus be ignored. However, when one is measuring a very small sample resistance, especially under variable temperature conditions, the contact resistance can dominate and completely obscure changes in the resistance of the sample itself.

The way out of this problem is to use the four point probe technique, which elimi- nates the effects of contact resistance. A schematic of a four point probe is shown in figure 2. In this diagram, four wires (or probes) have been attached to the test sample. A constant current is made to flow the length of the sample through probes labeled 1 and 4 in the figure. This can be done using a current source or a power supply as shown. Many power supplies have a current output readout built into them. If not, a digital ammeter, or even another voltmeter can be used to obtain the value of the current. A power supply capable of producing between 0.5 Amp to 1.0 Amp is required for the superconductor four point probe device in the kit.

If the sample has any resistance to the flow of electrical current, then there will be a drop of potential (or voltage) as the current flows along the sample between the two wires (or probes) labeled 2 and 3 in the figure. The voltage drop between probes 2 and 3 can be measured by a digital voltmeter. The resistance of the sample between probes 2 and 3 is the ratio of the voltage registering on the digital voltmeter to the value of the output current of the power supply. The high impedance of the digital voltmeter minimizes the current flow through the portion of the circuit comprising the voltmeter. Thus, since there is no potential drop across the contact resistance associated with probes 2 and 3, only the resistance associated with the sample between probes 2 and 3 is measured.

PROCEDURE

The four point probe device is encapsulated in a brass jacket to protect the super- conductor from mechanical and thermal stresses. The device has 6 wires. Four of these wires form the probes and are described in the next section. The red and blue pair of wires is connected to a low temperature thermocouple embedded in the super- conductor incorporated in the four point device. This thermocouple can be used to determine the temperature of the superconductor. A characterization table for this thermometer is included in the instruction manual.
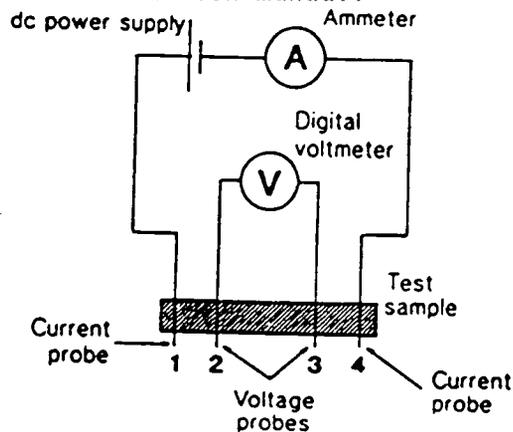

Figure 2 Schematic of a four point probe.

Measuring Tc and the effects of temperature on device resistance

This experiment and the measurement of Hc and Ic use the superconducting four point probe device described in this section. This experiment measures the change in resistance of the superconductor with a change in its temperature. As a part of this experiment, the critical temperature, Tc, of the superconductor is also obtained.
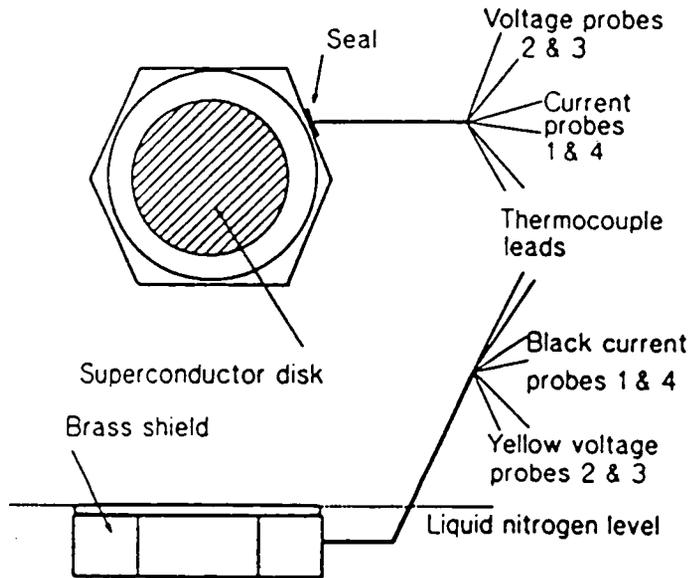

Figure 3   The superconducting four point probe configuration.

The four point probe device with its thermocouple is shown in figure 3. The pair of black wires are the current source probes, and are labeled 1 and 4 in figure 2. The pair of yellow wires are the voltage measurement probes, and are labeled 2 and 3 in figure 2. The paired blue and red wires are attached to the thermocouple junction in the device. During the experiment, the four point device is laid flat in the shallow dish in the kit so that the black disk superconductor is on the top, and the red plastic seal is at the bottom. The 6 wires are carefully bent so that they extend above the device and out of the liquid nitrogen as shown in figure 3. When liquid nitrogen is poured over the device, it should cover the superconductor disk, but the ends of the wires should project above the surface of the liquid. All electrical connections should be made above the surface of the liquid.

The voltmeters listed in the section entitled, "Equipment and Supplies", should be connected as shown in figure 2. Alternatively, a strip chart recorder with a 10 millivolt full-scale range and a resolution of 10 microvolts may be connected between probes 2 and 3. This will provide a continuous record of the voltage drop. If a two-channel recorder or x-y plotter is used, then the thermocouple reading can also be measured simultaneously. The current source or power supply should be set to produce a current of about 0.5 amp through probes 1 to 4.

The following is a step by step guide for measuring the device resistance versus its temperature:

1. Set up equipment as described in the last paragraph, but do not yet pour any liquid nitrogen over the device. The voltage drop between probes 2 and 3 should be between 2 to 5 mV.

2. Carefully pour liquid nitrogen over the device until it is just submerged.

3. When the device has completely cooled (as determined by the thermocouple reading), use the tweezers to remove the device from nitrogen and set it on the bench top. The device will start to warm up. The thermocouple reading will indicate this.

    Alternatively, the device can be allowed to warm as the liquid nitrogen evaporates.

4. As the device warms to above its critical temperature, the voltage drop across probes 2 and 3 will abruptly increase. The temperature at which this abrupt increase occurs is the critical temperature of the superconductor.

The ratio of the voltage between probes 2 and 3 to current flowing between probes 1 and 4 is the instantaneous resistance of the superconductor between probes 2 and 3. If this resistance is plotted versus the thermocouple reading, a result similar to that in figure 4 will be obtained. A strip chart recorder will much more conveniently record this result.

SAMPLE DATA
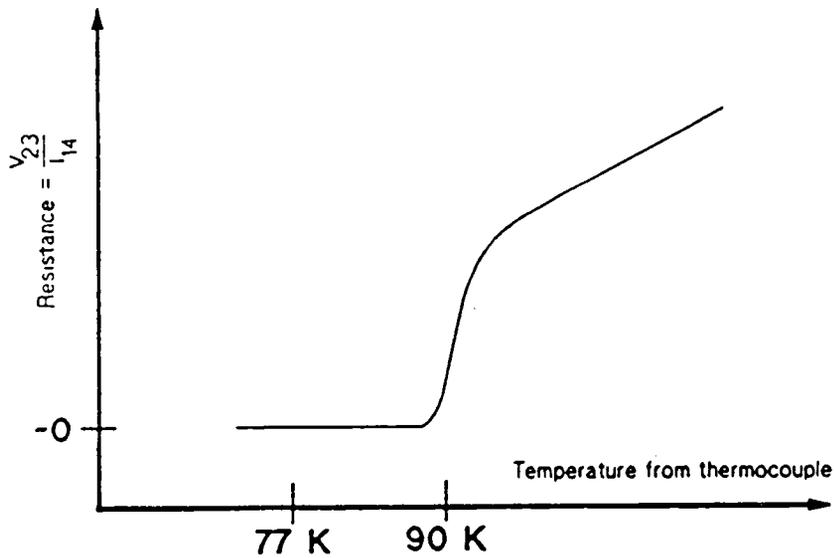
Figure 4 Graph of resistance variation with temperature.

Some Questions

1. What effect would one expect if the critical temperature is
    measured with the device placed inside a functioning
    electromagnet?

2. Why is the transition in resistance gradual at the critical
    temperature?

3. A simple two probe measurement of device resistance below its
    critical temperature exhibits a non-zero value. Why?

Measuring the Critical Magnetic Field, Hc

This experiment measures the critical magnetic field Hc, of a 1-2-3 YBCO superconductor using the four point probe device in the University Demonstration Kit.

For this experiment you will need a magnetic coil and associated power supply. The value of the field can be obtained using the geometry of the coil and a knowledge of the current flowing through it. The cavity in the middle of the coil needs to be large enough to accommodate the four point device and the liquid nitrogen container in which it is immersed. To ensure accuracy during this experiment, the four point probe device has been designed with no magnetic parts.

Assemble the experiment as for the measurement of the critical temperature Tc. Place the four point device and its container of liquid nitrogen inside the cavity of the electromagnet coil. Increase the current flowing through the electromagnet coil thus increasing the magnetic field strength at the superconductor. The signal between the voltage probes 2 and 3 will show an abrupt increase at some value of applied magnetic field strength. This value of magnetic field is the critical magnetic field Hc, for the YBCO superconductor sample at the temperature of liquid nitrogen, 77 Kelvin.

The value of the critical field H_c, can be obtained at other temperatures by either placing the device in a cryostat while performing this experiment, or by removing the device from the liquid nitrogen container and monitoring the output of the thermocouple thermometer while measuring Hc.

Another interesting experiment is to measure the critical temperature at different applied magnetic values. The result of such an experiment is shown schematically in figure 5. The value of Hc has been extrapolated to absolute zero temperature.


SAMPLE DATA
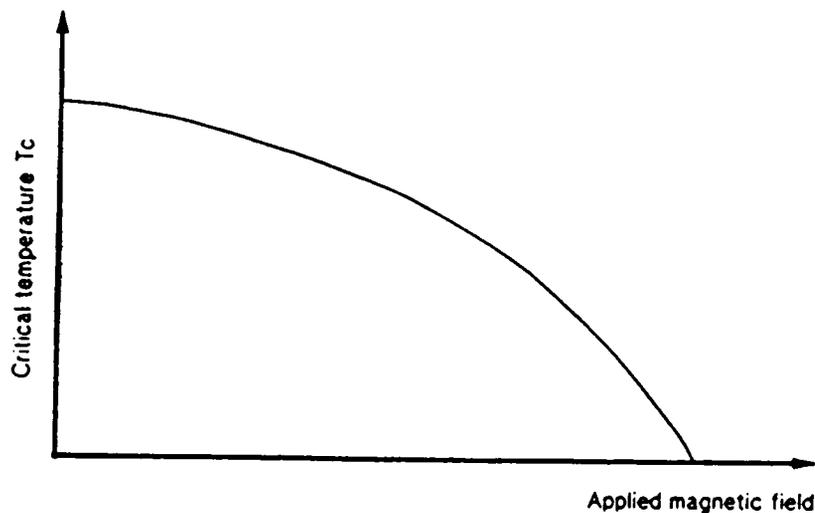
Figure 5 Variation of Critical Field with operating temperature.

Measuring the Critical Current, Ic, and the Current Density

The four point device can be used to measure the critical current Ic, and also the maximum current density of the 1-2-3 YBCO superconductor. However, since the value of current needed for this experiment is so large, great care must be exercised during its execution. In fact, if the instructions are not followed exactly, or the high current is left flowing through the device for an extended period of time, it could be severely damaged.

For this experiment, a power supply capable of putting out a current of about 15 amp across a resistance of about 1 ohm is needed. Connect the device to the digital voltmeters and power supply as explained in the section for measuring the critical temperature Tc. Here however, conduct the entire experiment while the device is submerged under liquid nitrogen. The liquid nitrogen serves the additional function of keeping the device and the connecting wires cool. The wires themselves are only marginally able to carry the large electrical current values needed. So, they too must be kept submerged in the liquid nitrogen.

When the two voltmeters are set, gradually increase the current through probes 1 and 4 while monitoring the voltage drop between probes 2 and 3. When the current value through probes 1 and 4 exceeds the critical current value for the superconductor, the voltage drop between probes 2 and 3 will abruptly increase. We have found that the critical current is about 10 amp for the device configuration used. This is the value of Ic at the 77 Kelvin temperature of liquid nitrogen.

Alternatively, one can measure the change in Tc at different values of current through the current probes 1 and 4. This, however, necessitates the removal of the device from liquid nitrogen which in turn has potential for damaging the device.

The critical current density can be obtained by a geometric analysis of the current flow through the device for the value of the critical current. For this analysis the following information is of use. The sample diameter is 24 mm. The distance between probes 2 and 3 is 11 mm, and probes 1 and 4 is 17.5 mm. The sample is 4 mm thick.

The Reverse ac Josephson Effect

In 1962 Brian Josephson showed that if there is a very thin electrically insulating film between two pieces of superconductor, then contrary to expectations, there will continue to be a flow of electrical current right through the insulator. This superconducting current is called a supercurrent. This phenomenon can only be explained by the esoteric field of quantum mechanics.

The new superconducting materials, like the one in this kit, are very granular. The individual grains are believed to be superconductors. But, each grain is separated from the next by what scientists think is a very thin film of insulator. One would then expect the same Josephson effect through these thin insulting films too. In the terminology of the new ceramic superconductors, these multiple Josephson junctions are called 'weak links'.

One way to actually observe the Josephson supercurrent is to perform the following experiment. In this experiment, a high frequency ac current is imposed on the superconductor. One will then observe a dc voltage across the superconductor along the same direction as the applied ac signal. This is called the reverse ac Josephson effect.

For this experiment one will need an ac signal source with a variable frequency capable of up to about 20 MHz and a peak to peak output of about 10 Volts, appropriate connecting wires, and a good voltage measurement system.

Connect the ac signal source to the current probes 1 and 4 of the four point probe device. Attach a digital voltmeter set for measuring dc voltage in the millivolt range between probes 2 and 3 of the device. The measured dc potential between probes 2 and 3 varies between 1 to 2 millivolts with a peak response at an applied signal of frequency 1 to 3 MHz. This result is shown schematically in figure 6.

Another interesting result is that if one approaches the surface of the superconductor disk with a magnet (for example the rare earth magnets in the kit), a decrease in the dc signal level is seen.

The experiment has the following very significant features:

1. It is a macroscopic demonstration of a quantum mechanical approach.

2. It exhibits the fundamental basis of the Josephson effect. This effect is expected to be of great importance in building the superfast computers of the future.

3. It is a very sensitive way to show that a material is superconducting. Scientists from Wayne State University used it to show that some of these materials may have a superconducting phase at temperatures at high as 240 Kelvin.

SAMPLE DATA
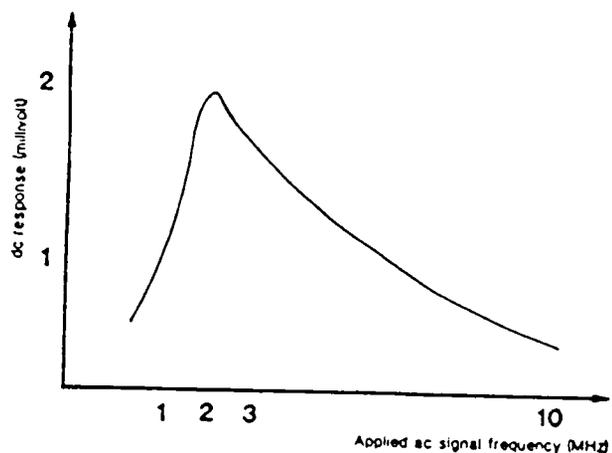
Figure 6   Plot shows the dc voltage as function of ac frequency.

INSTRUCTOR'S NOTE

Precautions

1.   When pouring liquid nitrogen, be careful to prevent
any splashing. Read the section in the manual on
safety and handling (pages 10, 11, 28, & 29) before
beginning the experiment.

2.   Conduct the experiments in a well-ventilated room.

3.   Do not touch any items immersed in the liquid nitro-
gen with your hand until they have warmed to room
temperature.

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